FINAL UNIFORM FEDERAL POLICY QUALITY ASSURANCE PROJECT PLAN FOR SITE INSPECTION

PERFORMANCE BASED REMEDIATION TASK ORDER PETERSON AIR FORCE BASE, SITES OW011 and OW012

Contract No: FA8903-09-D-8578

Task Order: 0003

SubCLINs: 0009EV and 0009EX

March 2013

Revision 2



Air Force Civil Engineer Center 2261 Hughes Avenue, Suite 155 Lackland AFB, Texas 78236-9853

Prepared by



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in association with



VERSAF



EXECUTIVE SUMMARY

Site Location and History

Peterson Air Force Base (AFB) is located on the eastern border of Colorado Springs, El Paso County, Colorado, and provides runways for the adjacent City of Colorado Springs Municipal Airport under a shared joint civil-military airport agreement. The installation is approximately 1,278 acres in size and is at an elevation of 6,035 feet above mean sea level (msl).

Peterson AFB began as the Colorado Springs Army Air Base, established on April 28, 1942, at the Colorado Springs Municipal Airport, which had been in operation since 1926. The installation carried out photo reconnaissance training under the auspices of the Photo Reconnaissance Operational Training Unit. On June 22, 1942, the Colorado Springs Army Air Base was assigned to the 2nd Air Force, headquartered at Fort George Wright, Washington. On December 13, 1942, officials changed the name of the Colorado Springs Army Air Base to Peterson Army Air Base, in honor of 1st Lieutenant Edward Joseph Peterson, who was killed in a crash at the installation. The installation assumed a new mission of heavy bomber combat crew training in October 1943. On December 31, 1945, the Army inactivated the installation and turned the property over to the City of Colorado Springs. The installation was then reactivated three times: from September 29, 1947, to January 15, 1948; from September 22, 1948, into 1949; and following the January 1951 establishment of Air Defense Command. On March 1, 1976, Peterson Field was renamed Peterson AFB. Strategic Air Command assumed control of the installation on October 1, 1979. On September 1, 1982, control was transferred from Strategic Air Command to the newly established Air Force Space Command, which was activated at Peterson AFB the same day.

Principal military flight operations at Peterson AFB are currently conducted by the 302^{nd} Airlift Wing, an Air Mobility Command-gained unit of the Air Force Reserve Command (Peterson AFB, 2007). Currently, land use at Peterson AFB consists of a combination of administrative, industrial, special space mission, residential, community, recreational, and aircraft operations. Industrial operations have generally been associated with aircraft and vehicle maintenance such as engine repair, aircrafts systems maintenance, and painting (URS 2010). Peterson AFB employs approximately 18,000 military personnel, Department of Defense (DoD) civilians, and contractors (Peterson AFB 2009).

Description and Previous Investigation of Site OW011

Site OW011 at Peterson AFB consists of an abandoned concrete oil/water separator (OWS) (approximately 4 feet [ft] by 4 ft by 9.5 ft deep) that previously received floor wash water from floor drains; the water may have contained petroleum, oil, and lubricants (POL). A 2008 report noted the vault beneath the sewer covers had been filled in with concrete to a depth of approximately 4 inches below ground surface (bgs); however, no information regarding actions taken to prevent discharges to and from the OWS was available.

Soil samples were collected in April 11, 2012, during an investigation of the OWS to determine Defense Environmental Remediation Account (DERA) eligibility. Soil samples collected from the anticipated OW011 influent pipe line were selected for laboratory analyses based on high photoionization detector (PID) readings and were analyzed by an off-site laboratory for volatile organic compounds (VOCs), total petroleum hydrocarbon–gasoline range organics (TPH-GRO), and TPH-diesel range organics (DRO). The April 2011 DERA Investigation Report concluded that the one soil sample identified a potential release from the OWS, based on the TPH-DRO concentration of 2,400 milligrams per kilogram (mg/kg); therefore, the other two samples were not analyzed, consistent with the DERA protocols. There were also several detections for VOCs at Site OW011 including one exceedance for naphthalene compared to the EPA regional screening level for residential soil (RSL-R).

Summary of Sampling Plan for Site OW011

Additional soil samples will be collected from Site OW011 as part of this Site Inspection (SI) to confirm and then further delineate the contamination found during the 2011 DERA investigation, if necessary. Delineation samples may be collected from within 5 lateral ft of the trench drain effluent, OWS influent, and OWS effluent, as presented in Figure 17-1 of this Uniform Federal Policy-Quality Assurance Project Plan (UFP-QAPP). Soil samples are anticipated to be collected using direct-push technology. In situations where direct push is not possible, soil samples will be collected using hand augers, shovels or equivalent methods. Additional step-out and step-down delineation samples may be collected at intervals of no more than 5 ft laterally and 2 ft vertically. All soil samples collected will be screened with a PID and any visual staining and/or hydrocarbon odor observed will be noted in a field logbook. Step-out and step-down delineation sampling will continue until PID readings, visual staining, and hydrocarbon odor have diminished. Additional, non-biased samples may be collected to support risk-based, exposure point concentration (EPC) calculations. Samples will be analyzed for VOCs and semi-volatile organic compounds (SVOCs), which represents compounds that were either detected during the 2011 DERA investigation or may be present based on the known site history.

Description and Previous Investigations of Site OW012

Site OW012 at Peterson AFB includes an area near Hole 2 at the Peterson AFB Golf Course. The site is within the boundaries of a former Installation Remediation Program (IRP) site that contained a leach field system. The former leach field consisted of a settling tank (likely the OWS for Site OW012) and a gravel-envelope drainage field. A Remedial Investigation (RI) for the former IRP site was completed in 1989. Data and information collected during the RI demonstrated no adverse impact to human health or the environment, based on a risk assessment, and it was determined that no further action was necessary. Based on those findings, the site was eliminated from further consideration under the IRP.

A 2008 report indicated magnetic anomalies were observed at two locations approximately 140 ft south of Hole 2 and 30 ft east of a north/south golf cart road. The April 2011 DERA investigation sampling locations were based on this information and personnel interviews indicating that a formerly active OWS may be located in this area. The estimated location of Site OW012 is within the footprint of the leach field settling tank as depicted in the 1989 RI report. For the 2011 DERA investigation, three borings were drilled approximately 50 ft apart around Site OW012 (based on the magnetic anomalies), and one sample was collected from each boring. The only sample analyzed at the laboratory was collected approximately 100 ft east of the OWS; naphthalene was detected above the EPA RSL-R in this sample.

Summary of Sampling Plan for Site OW012

Additional soil samples will be collected from Site OW012 as part of this SI to confirm and then further delineate the contamination found during the 2001 DERA investigation, if necessary. Delineation samples may be collected from within 10 lateral ft of the sample locations identified in the 2011 DERA investigation, as presented in Figure 10-3 of the UFP-QAPP. Soil samples will be collected using direct-push technology, hand augers, and/or shovel, as appropriate. Additional step-out and step-down delineation samples may be collected at intervals of no more than 5 ft laterally and 2 ft vertically. Samples collected will be screened with a PID and any visual staining and/or hydrocarbon odor observed will be noted in a field logbook. Step-out and step-down delineation sampling will continue until PID readings, visual staining, and hydrocarbon odor have diminished to non-detect, if present. Additional, non-biased samples may be collected to support risk-based EPC calculations based on EPA Region 8 guidelines. Samples will be analyzed for VOCs and SVOCs, which represents compounds that were either detected during the 2011 DERA Investigation or may be present based on the previous site history.

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APPENDIX B - Project Standard Operating Procedures

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ATTACHMENTS

ATTACHMENT 1 – Site Safety and Health Plan

ACRONYMS

°C degrees Centigrade
AFB Air Force Base

AFCEC Air Force Civil Engineer Center

BA Bachelor of Arts
Bgs below ground surface
BS Bachelor of Science
CA Corrective Action

CCV continuing calibration verification

CDPHE Colorado Department of Public Health and Environment

CFR Code of Federal Regulations
CIH Certified Industrial Hygienist

COC chain of custody

COI Contaminant of Interest
CSM Conceptual Site Model

DERA Defense Environmental Remediation Account

DL detection limit

DoD Department of Defense
DPT direct push technology
DQI data quality indicators
DQO data quality objective

ECC Environmental Chemical Corporation
EDMS environmental data management system

EDD electronic data deliverable

e.g. for example

ELAP Environmental Laboratory Program
EPA Environmental Protection Agency
EPC Exposure Point Calculation

ERA Environmental Restoration Account

ft feet/foot

GC gas chromatograph

HAZWOPER Hazardous Waste Operations

ICAL Initial Calibration i.e. in other words

IRP Installation Remediation Program

J estimated

LCS laboratory control sample
LOQ level of quantitation
μg/kg micrograms per kilogram
mg/kg Milligrams per kilogram

MPC measurement performance criteria

MS Master of Science for education (WS #4,7 & 8)

MS mass spectrometer for equipment type (instrumentation, WS #23)

MS/MSD matrix spike/matrix spike duplicate

msl mean sea level

MQO Measurement Quality Objective

ACRONYMS

NA Not Applicable
NFA No Further Action

OSHA Occupational Safety and Health Administration

OWS oil/water separator
PAL project action level

PBR Performance Based Remediation
PDF portable document format

PDF portable document format
PE Professional Engineer
PG Professional Geologist
PID photo ionization detector

PM Project Manager

PMP Project Management Professional POL petroleum, oil, and lubricants

PT proficiency testing
QA quality assurance

QAM Quality Assurance Manager

QC quality control

QCM Quality Control Manager
QSM Quality Systems Manual

RF response factor

RI Remedial Investigation

RL reporting limit

RPD relative percent difference

% **RSD** percent relative standard deviation

RSL Regional Screening Level

RMA-Insight RMA-Insight Engineering and Construction, JV

SB soil boring
SI Site Inspection

SOP Standard Operating Procedure

SPCC system performance check compound SVOC semi-volatile organic compounds

SW solid waste

SWMU solid waste management unit

TA TestAmerica – Denver

TCE Trichloroethene
TO Task Order

TPH total petroleum hydrocarbon

TPH- DRO total petroleum hydrocarbon-diesel range organics
TPH-GRO total petroleum hydrocarbon-gasoline range organics

TSA Technical Systems Audit
UJ estimated non-detect

UFP-QAPP Uniform Federal Policy-Quality Assurance Project Plan

USACEU.S. Army Corps of EngineersVOCvolatile organic compounds

WS Worksheet

Table 1. Crosswalk: UFP-QAPP Workbook to 2106-g-05 QAPP

Optimiz	Optimized UFP-QAPP Worksheets 2106-G-05 QAPP Guidance Section				
1 and 2	Title and Approval Page	2.2.1	Title, Version, and Approval/Sign-Off		
3 and 5	Project Organization and QAPP	2.2.3	Distribution List		
3 una 3	Distribution		Project Organization and Schedule		
4,7	Personnel Qualifications and Sign-off	2.2.4	Title, Version, and Approval/Sign-Off		
and 8	Sheet	2.2.7	Special Training Requirements and Certification		
6	Communication Pathways	2.2.4	Project Organization and Schedule		
9	Project Planning Session Summary	2.2.5	Project Background, Overview, and Intended Use of Data		
10	Conceptual Site Model	2.2.5	Project Background, Overview, and Intended Use of Data		
11	Project/Data Quality Objectives	2.2.6	Data/Project Quality Objectives and Measurement Performance Criteria		
12	Measurement Performance Criteria	2.2.6	Data/Project Quality Objectives and Measurement Performance Criteria		
13	Secondary Data Uses and Limitations	Chapter 3	QAPP Elements for Evaluating Existing Data		
14 and 16	Project Tasks and Schedule	2.2.4	Project Organization and Schedule		
15	Project Action Limits and Laboratory- Specific Detection / Quantitation Limits	2.2.6	Data/Project Quality Objectives and Measurement Performance Criteria		
17	Sampling Design and Rationale	2.3.1	Sample Collection Procedure, Experimental Design, and Sampling Tasks		
18	Sampling Locations and Methods	2.3.1	Sample Collection Procedure , Experimental Design, and Sampling Tasks		
		2.3.2	Sampling Procedures and Requirements		
19 and 30	Sample Containers, Preservation, and Hold Times	2.3.2	Sampling Procedures and Requirements		
20	Field QC	2.3.5	Quality Control Requirements		
21	Field SOPs	2.3.2	Sampling Procedures and Requirements		
22	Field Equipment Calibration, Maintenance, Testing, and Inspection	2.3.6	Instrument/Equipment Testing, Calibration and Maintenance Requirements, Supplies and Consumables		

Table 1. Crosswalk: UFP-QAPP Workbook to 2106-g-05 QAPP

Optimiz	zed UFP-QAPP Worksheets	2106-G-05 QAPP Guidance Section		
23	Analytical SOPs	2.3.4	Analytical Methods Requirements and Task Description	
24	Analytical Instrument Calibration	2.3.6	Instrument/Equipment Testing, Calibration and Maintenance Requirements, Supplies and Consumables	
25	Analytical Instrument and Equipment Maintenance, Testing, and Inspection	2.3.6	Instrument/Equipment Testing, Calibration and Maintenance Requirements, Supplies and Consumables	
26 and 27	Sample Handling, Custody, and Disposal	2.3.3	Sample Handling, Custody Procedures, and Documentation	
28	Analytical Quality Control and Corrective Action	2.3.5	Quality Control Requirements	
29	Project Documents and Records	2.2.8	Documentation and Records Requirements	
31, 32, and 33	Assessments and Corrective Action	2.4	Assessments and Data Review	
		2.5.5	Reports to Management	
34	Data Verification and Validation Inputs	2.5.1	Data Verification and Validation Targets and Methods	
35	Data Verification Procedures	2.5.1	Data Verification and Validation Targets and Methods	
36	Data Validation Procedures	2.5.1	Data Verification and Validation Targets and Methods	
37	Data Usability Assessment	2.5.2	Quantitative and Qualitative Evaluations of Usability	
		2.5.3	Potential Limitations on Data Interpretation	
		2.5.4	Reconciliation with Project Requirements	

Notes:

QC – quality control

SOP – Standard Operating Procedure

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QAPP Worksheet #1 and 2: Title and Approval Page (UFP-QAPP Manual Section 2.1) (EPA 2106-G-05 Section 2.2.1)

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- a. Site name/project name: Performance-Based Remediation Task Order (TO) for Peterson AFB, Sites OW011 and OW012
- b. Site location/number: Peterson AFB, Colorado Springs, Colorado, Sites OW011 and OW012

		0 11 012
	c.	Contract/Work Assignment Number: FA8903-09-R-9999-R154, TO 003, SubClins 0009EV and 0009EX
2.	Lead O	rganization
	a.	Lead Organization Project Manager (name/title/signature/date)
		Robert Fant, Chief, Environmental Quality, Peterson AFB
	b.	Lead Organization Project Manager (name/title/signature/date)
		Sharon Stone, Environmental Restoration Account (ERA) Project Manager, Peterson AFB
	c.	Lead Organization Project Manager (name/title/signature/date):
		Ernesto, Perez, Air Force Civil Engineer Center (AFCEC)-EX
2	Es demal	Deculators Acares (remarkitle/signature/date)
Э.	rederai	Regulatory Agency (name/title/signature/date)
		Dave Rathke, Project Manager, Environmental Protection Agency (EPA), Region 8
4.	State Re	egulatory Agency (name/title/signature/date)
		Lee Pivonka, State Project Officer, Colorado Department of Public Health and Environment (CDPHE)

5. List plans and reports from previous investigations relevant to this project:

FINAL Technical Evaluation Report, Investigation of OWSs to Determine DERA Eligibility, Peterson Air Force Base, July 2011 (URS, 2011)

Silver Spruce Golf Course, Environmental Management Plan, Peterson AFB, Colorado, June 2008

FINAL Decision Document for Leach Field, Peterson Air Force Base (Science Applications International Corporation October 1989)

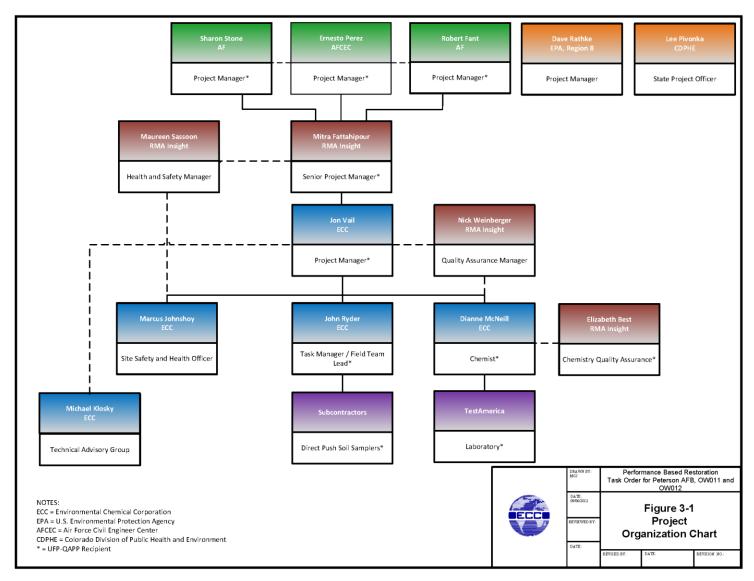
FINAL Remedial Investigation Report, Peterson Air Force Base (Science Applications International Corporation September 1989)

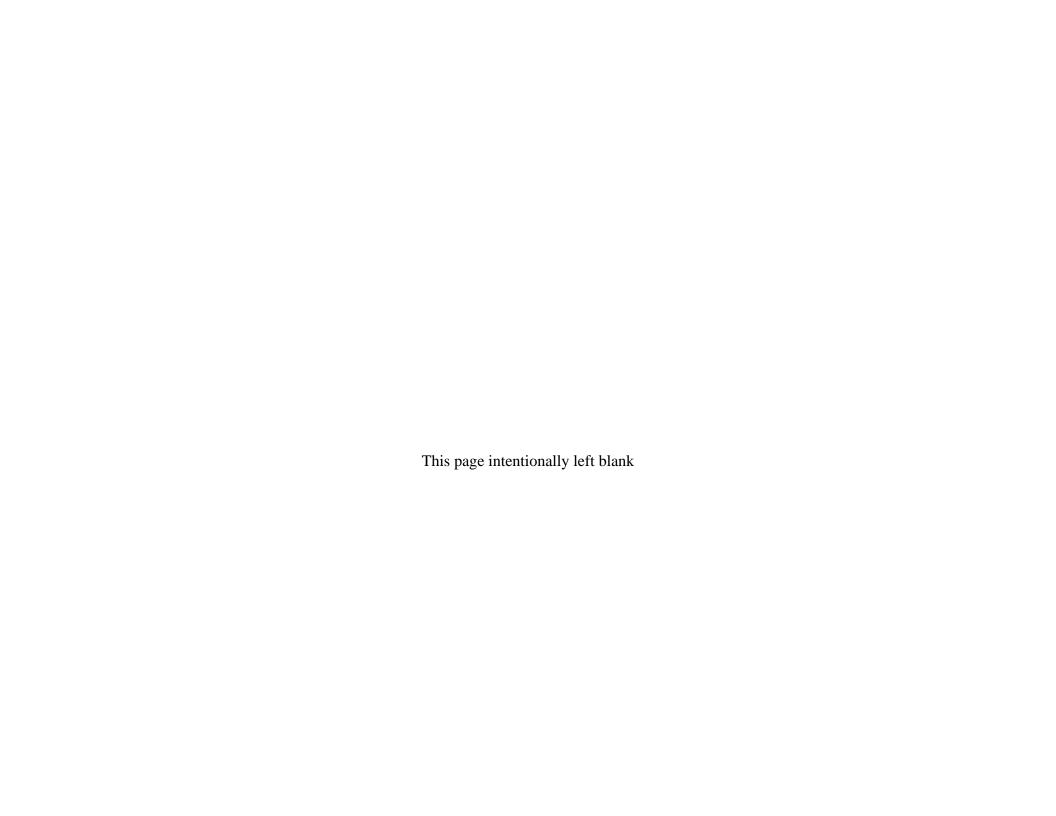
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QAPP Worksheet #3 and 5: Project Organization and QAPP Distribution (UFP-QAPP Manual Section 2.3 and 2.4) (EPA 2106-G-05 Section 2.2.3 and 2.2.4)

*QAPP recipient	Lines of authority	Lines of Communication

Figure 3-1: Organizational Chart





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QAPP Worksheet #4, 7 and 8: Personnel Qualifications and Sign-Off Sheet (UFP-QAPP Manual Sections 2.3.2 – 2.3.4) (EPA 2106-G-05 Section 2.2.1 and 2.2.7)

ORGANIZATION: RMA-Insight Engineering and Construction, JV (RMA-Insight)

Name	Project Title/Role	Education/Experience	Specialized	Signature/Date
			Training/Certifications	
Mittra Fatthipour	Senior Project Manager	Bachelor of Science (BS)	Professional Geologist	
	(PM)	Geology, 25 years'	(PG)	
		experience		
Nick Weinberger	QC Manager (QCM)	BS Chemistry, 15 years'		
		experience		
Maureen Sassoon	Safety and Health Manager	Master of Science (MS)	Certified Industrial	
		Environmental &	Hygienist (CIH)	
		Occupational Health,		
		Master of Public Health,		
		Doctorate in Public		
		Administration, >30 years'		
		experience		
Elizabeth Best	Program Chemist	BS Marine Science, 6		
		years' experience		

ORGANIZATION: Environmental Chemical Corporation (ECC)

Name	Project Title/Role	Education/Experience	Specialized Training/Certifications	Signature/Date
Jon Vail	PM	BS Geology, 21 years' experience	PG, Project Management Professional (PMP) 40- Hour Hazardous Waste Operations (HAZWOPER), Occupational Safety and Health Administration (OSHA) 29 Code of Federal Regulations (CFR) 1910.120 w/annual refresher	
John Ryder	Task Manager	BS Environmental Biology, Chemistry Minor, 12 years' experience	40-Hour HAZWOPER w/annual refresher, OSHA 30-Hour Construction Safety and Health	
Michael Klosky	Technical Advisory Group	BS Chemical/ Environmental Engineering, 22 years' experience	Professional Engineer (PE), HAZWOPER w/annual refresher	
Dianne McNeill	Project Chemist	Bachelor of Arts (BA) Biology/German, General Science (Physics/Chemistry) Minor, 19 years' experience	40-Hour HAZWOPER, OSHA 29 CFR 1910.120 w/annual refresher; 8-Hour HAZWOPER Supervisor Training	

Name	Project Title/Role	Education/Experience	Specialized	Signature/Date
	_	_	Training/Certifications	_
Marcus Johnshoy	Site Safety and Health	MPH Environmental	Certified Industial	
	Officer	Health, MS Microbiology,	Hygienist; Certified Safety	
		BS Microbiology, 20	Professional; U.S. Army	
		years' experience	Corps of Engineers	
			(USACE) 40-Hour	
			Engineering Manual 385-	
			1-1 Safety, 2012;	
			OSHA Construction	
			Industry Outreach Trainer	
			Update #502, 2008;	
			OSHA 30-Hour	
			Construction Safety and	
			Health; Scaffolding Safety	
			Fundamentals; 40-Hour	
			HAZWOPER Training,	
			Refresher, 2011; 8-Hour	
			HAZWOPER Supervisor	
			Training Safety	
			Management, Part I and II,	
			ASSE, 2003; First Aid/	
			Cardiopulmonary	
			Resuscitation, 2011	

ORGANIZATION: Laboratory

Name	Project Title/Role	Education/Experience	Specialized	Signature/Date
			Training/Certifications	
Deb Henderer	Laboratory Client Services	Not Applicable (NA)	NA	
	Manager, TestAmerica -			
	Denver			

^{*}Signatures indicate personnel have read and agree to implement this QAPP as written

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QAPP Worksheet #6: Communication Pathways (UFP-QAPP Manual Section 2.4.2) (EPA 2106-G-05 Section 2.2.4)

Communication Driver	Organization	Name	Contact Information	Procedure (timing, pathway, documentation, etc.)
Contractual updates including milestones and schedule	AFCEC	Ernesto Perez AFCEC PM	ernesto.perez@us.af.mil 307-773-3468	Monthly reports, including schedule updates are subject to AFCEC PM review. PM for AFCEC will be copied on all project correspondence with the PM and Regulators.
Regulatory agency interface	Peterson AFB	Robert Fant PM and/or Sharon Stone PM	robert.fant.1@us.af.mil 719-556-6100 sharon.stone@us.af.mil 719-554-5819	All materials and information about the project will be forwarded to the PM by RMA-Insight's PM or the ECC Task Manager (with RMA-Insight PM copied). The PM is the main point of contact for the project.
Contractor project management needs	RMA-Insight	Mitra Fattahipour Prime Contractor PM	mfattahipour@ieeci.com 858-342-5585	Overall project management actions.
Field progress reports	ECC	John Ryder ECC PM	jryder@ecc.net 720-232-6425	Maintenance of lines of communication between AFCEC, Peterson AFB, EPA Region 8, CDPHE, and subcontractors

Communication Driver	Organization	Name	Contact Information	Procedure (timing, pathway, documentation, etc.)
Stop work due to safety issues	Peterson AFB/ECC	Robert Fant, Sharon Stone or delegates, and Marcus Johnshoy	robert.fant.1@us.af.mil 719-556-6100 sharon.stone@us.af.mil 719-554-5819 MJohnsoy@ecc.net 303-887-7427	All employees on site have stop- work authority. Bob Fant and Sharon Stone (Peterson AFB) and Marcus Johnshoy (ECC) have overall authority of safety on site. Marcus has responsibility for ensuring all Contractor team employees and subcontractors
QAPP Implementation	ECC	John Ryder ECC PM	jryder@ecc.net 720-232-6425	follow applicable safety plans. Oversight of maintaining and implementing approved QAPP. Overall responsibility for distribution of QAPP revisions.
QAPP changes prior to field work	ECC	John Ryder ECC PM	jryder@ecc.net 720-232-6425	QAPP changes following QAPP approval will be documented and reasons for the change will be clearly communicated to the client by the designated Point of Contact (in other words [i.e.], the ECC PM will contact the AFCEC PM) within 24 hours of receipt of information justifying the change. All QAPP changes are subject to AFCEC PM and Peterson AFB PMs approval.
QAPP changes during project execution	ECC	John Ryder ECC PM Field Team Lead	jryder@ecc.net 720-232-6425	Notifies ECC PM or designee by phone. E-mail changes to QAPP and reasons to RMA-Insight QCM immediately.

				Procedure
Communication Driver	Organization	Name	Contact Information	(timing, pathway, documentation,
				etc.)
Field corrective actions	RMA-Insight	Nick Weinberger	NWeinberger@ieeci.com	The QCM will determine the need
		Contractor QCM	714-678-6700	for field corrective actions. Any
				corrective actions will be
				communication to the AFCEC PM
				and Peterson AFB PMs within 24
				hours.
Technical Advisement	ECC	Michael Klosky	mklosky@ecc.net	Provide overall project support in
		Technical	770-889-6277	the role of advisor.
		Advisor		
Field Implementation	ECC	John Ryder	jryder@ecc.net	Prepare Daily Quality Control
		ECC PM	720-232-6425	Reports; implement Final UFP-
		Field Team Lead		QAPP in the field; identify field
				discrepancies; prepare all field
				documentation
Sample receipt variances	Test-America	Deb Henderer	Debra.Henderer@testamericainc	The laboratory PM will
	Denver (TA)		<u>.com</u>	communicate any sample receipt
			303-736-0134	issues immediately via phone or e-
				mail to the Project Chemist. If the
				issue requires action, the Project
				Chemist will consult with the ECC
				PM, who will work with the QCM
				to address any related field issues.
Laboratory quality control	TA	Deb Henderer	<u>Debra.Henderer@testamericainc</u>	All quality assurance (QA)/QC
variances			. <u>com</u>	issues are reported to the Project
			303-736-0134	Chemist immediately. The
				contractor Project Chemist will
				work with the laboratory to identify
				the issue and possible solutions; this
				information will be immediately
				communicated to the ECC PM and
				Contractor QCM, who will contact
				the client.

Communication Driver	Organization	Name	Contact Information	Procedure (timing, pathway, documentation,
	3 -g			etc.)
Analytical corrective actions	TA	Deb Henderer	Debra.Henderer@testamericainc .com 303-736-0134	Analytical corrective actions will be made by laboratory personnel. Any action affecting data quality, turnaround time, or data reporting will be communicated by the laboratory analysts to the laboratory PM, who will in turn communicate will the Project Chemist via e-mail or phone immediately upon identification of the issue. All issues will be documented and communicated by the Project Chemist to the QCM and ECC PM immediately.
Data verification issues, for example (e.g.), incomplete records	ECC	Dianne McNeill Project Chemist	dmcneill@ecc.net 303-590-1182	Under the direction of the Project Chemist, data will be verified in as close to "real time" as possible. The Project Chemist will coordinate between the subcontract laboratory and project team to resolve any discrepancies.
Data validation issues, e.g., noncompliance with procedures	ECC	Dianne McNeill Project Chemist	dmcneill@ecc.net 303-590-1182	The Project Chemist will immediately contact the laboratory PM upon identification of noncompliant laboratory actions. The QCM will be notified of any laboratory noncompliance and the impact on project objectives within 24 hours. The QCM will be notified immediately upon identification of noncompliant field actions.

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				Procedure
Communication Driver	Organization	Name	Contact Information	(timing, pathway, documentation,
				etc.)
Data review corrective actions	RMA-Insight	Nick Weinberger	NWeinberger@ieeci.com	The Program Chemist will
		Contractor QCM	714-678-6700	determine the need for data review
				corrective actions. Any corrective
		Elizabeth Best		actions will be communication to
		Contractor Team	EBest@ieeci.com	the contractor QCM, who will
		Program Chemist	970-302-6084	evaluate the significance of the
				issue to the overall project. Issues
				affecting data that has been used for
				decisions in the field will be
				communicated by the QCM to the
				AFCEC PM and Peterson AFB PMs
				within 24 hours.

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QAPP Worksheet #9: Project Planning Session Summary (UFP-QAPP Manual Section 2.5.1 and Figures 9-12) (EPA 2106-G-05 Section 2.2.5)

Date of planning session: 06 September 2012 Location: Peterson Air Force Base (AFB)

Purpose: Project Kick-Off Peterson AFB and Schriever AFB

Participants:

Name	Organization	Title/Role	E-mail/Phone
Robert Fant	21 CES/CEAN	Chief, Environmental Quality	robert.fant.1@us.af.mil 719-556-6100
Sharon Stone	HQ AFSPS/A7M	ERA Project Manager (PM)	sharon.stone@ us.af.mil 719-554-5819
Albert Fernandez	50 CES/CEAN	Environmental Engineer	albert.fernandez.1@us.af.mil 719-567-4028
Kim Nemmers	ECC	Project Manager	knemmers@ecc.net 303-532-9132
David Cox	RMA-Insight	Project Manager	dcox@iecci.com 720-250-8551
John Ryder	ECC	Task Manager	<u>jryder@ecc.net</u> 720-232-6425

Notes/Comments:

David Cox, Program Manager from RMA-Insight, introduces himself, and then John Ryder, and Kim Nemmers from ECC. RMA-Insight is the prime contractor for this Performance Based Remediation (PBR) Task Order, and ECC is a subcontractor under RMA-Insight. Two OWS sites at Peterson AFB, and two OWS sites at Schriever AFB are contracted under this PBR.

Meeting attendees were provided with hard copies of a PowerPoint slide presentation. Material included background information on both AFBs, descriptions of OWS sites, sample results, and anticipated field activities.

The work plans for the project will be in UFP-QAPP format. The UFP-QAPP will contain the field sampling plan, all contact information, standard operating procedures (SOPs), etc., for the project. The approach for all OWS sites at Schriever AFB and Peterson AFB is a commitment to unrestricted site closure.

The 2011 OWS site investigation included 12 sites at Schriever AFB and 19 sites at Peterson AFB. Only 2 sites from each AFB are included in this PBR.

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Peterson AFB Site Discussion:

John Ryder began the discussion of the Peterson AFB sites with information on Site OW011, which included the history of Building 1322, its uses, and the likely wastes/products received by the OWS. Bob Fant commented on the history of the buildings and area that surround Building 1322 (Site OW011). Observing the picture of Building 1322, Mr. Fant indicated that he was the vehicle control manager and that the Building 1322 was used as the snow barn. The building behind Building 1322 was not present during that time. Mr. Fant stated that the snow plows and other heavy equipment were managed and cleaned up at the building.

The 1989-Phase I IRP at Peterson AFB included the area where the OW012 is located. Additionally, Mr. Fant indicated that conversations with the AFB personnel revealed that there was a release at the site in 1988, which was verbally reported. Mr. Fant provided information about the location of the OW012 at the Golf Course. The site is off the west end of the greens by at least 50 yards, with no access issues. No Federal Aviation Authority notifications or permits will be required.

The handout material provided contained information about photo ionization detector (PID) readings collected during the OWS investigations at Peterson AFB. Mr. Ryder explained that a PID is used in the field to detect volatile organic compounds. The PID readings collected in the field during 2011 were used to determine which samples would be analyzed at the laboratory. Kim Nemmers added that the reports indicate that hydrocarbon odor was likely noticeable from the samples. The process that the OWS investigation followed was to analyze only one sample if there were odor or high PID readings. The other samples remained on hold at the lab pending the results from the analyzed sample. If the initial sample indicated detection(s) of an analyte or compound, then the other two soil samples collected were not analyzed.

Sample results from Sites OW011 and OW012 showed detections of TPH-DRO (total petroleum hydrocarbon-diesel range organics) and TPH-GRO (gasoline range organics). TPH-DRO represents a heavier hydrocarbon range, which could contain diesel fuels, oil, and hydraulic fluids. TPH-GRO includes lighter fuels like gasoline. Only one soil sample was analyzed from each OWS site at Peterson AFB.

OWS Sites are not specifically regulated and the RMA-Insight Team asked if the regulators were going to be involved for work at Peterson AFB. The RMA-Insight Team suggested completion of the SI before involving the regulators to better understand the sites. Mr. Fant and Ms. Stone indicated that Regulatory involvement may be preferred earlier on for the Peterson AFB Sites and would follow up internally.

Action Items:

Site (Peterson)	Action	Responsible Party	Due Date
OW011	Decision on Regulatory	Sharon Stone and/or Bob Fant	October 12,
and	Involvement for		2012
OW012	Peterson Sites		

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QAPP Worksheet #10: Conceptual Site Model (UFP-QAPP Manual Section 2.5.2) (EPA 2106-G-05 Section 2.2.5)

10.1 Site History

Peterson AFB is located on the eastern border of Colorado Springs, El Paso County, Colorado, and provides runways for the adjacent City of Colorado Springs Municipal Airport under a shared joint civil-military airport agreement. The installation is approximately 1,278 acres in size and is at an elevation of 6,035 feet above msl.

Peterson AFB began as the Colorado Springs Army Air Base, established on April 28, 1942, at the Colorado Springs Municipal Airport, which had been in operation since 1926. The installation carried out photo reconnaissance training under the auspices of the Photo Reconnaissance Operational Training Unit. On June 22, 1942, the Colorado Springs Army Air Base was assigned to the 2nd Air Force, headquartered at Fort George Wright, Washington. On December 13, 1942, officials changed the name of the Colorado Springs Army Air Base to Peterson Army Air Base, in honor of 1st Lieutenant Edward Joseph Peterson, who was killed in a crash at the installation. The installation assumed a new mission of heavy bomber combat crew training in October 1943. On December 31, 1945, the Army inactivated the installation and turned the property over to the City of Colorado Springs. The installation was then reactivated three times: from September 29, 1947, to January 15, 1948; from September 22, 1948, into 1949; and following the January 1951 establishment of Air Defense Command. On March 1, 1976, Peterson Field was renamed Peterson AFB. Strategic Air Command assumed control of the installation on October 1, 1979. On September 1, 1982, control was transferred from Strategic Air Command to the newly established Air Force Space Command, which was activated at Peterson AFB the same day.

Principal military flight operations at Peterson AFB are currently conducted by the 302nd Airlift Wing, an Air Mobility Command-gained unit of the Air Force Reserve Command (Peterson AFB 2007). Currently, land use at Peterson AFB consists of a combination of administrative, industrial, special space mission, residential, community, recreational, and aircraft operations. Industrial operations have generally been associated with aircraft and vehicle maintenance such as engine repair, aircrafts systems maintenance, and painting (URS 2010). Peterson AFB employs approximately 18,000 military personnel, DoD civilians, and contractors (Peterson AFB 2009).

10.1.1 Site OW011

OW011 received floor wash water from drains of the Material Supply Shop (Building 1322). Water from the floor drains within Building 1322 entered an OWS in front of the building in the parking lot near the southwest wall of the building in front of Bays 2 and 3. Waste product was pumped from the OWS by a contracted used-oil recycler, and water was discharged from the OWS through an iron pipe to the sanitary sewer. No information regarding actions taken to prevent discharges to and from the OWS is available. No records are available to indicate sampling activities have occurred at Site OW011 site prior to the 2011 DERA investigation. Water entering the OWS may have contained POL.

10.1.1 Site OW012

Site OW012 is located within a former IRP site on the Base Golf Course. A preliminary assessment of potential environmental contamination at this site was conducted in 1985. Findings from the preliminary assessment recommended the area be investigated further, which resulted in a RI conducted in 1989. The

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site is within a former leach field system that consisted of a settling tank and an OWS, and a gravel envelope drainage (leach) field. Influent to the system came from the industrial area waste line. Beginning in 1956, this system received the majority of industrial liquid waste, including solvents, paint and paint residues, strippers, and waste oils from the AFB.

10.2 Previous Investigations

An investigation of OWSs to determine DERA eligibility was conducted at Peterson AFB in April 2011. During this investigation samples were collected from Sites OW011 and OW012. TPH-DRO, TPH-GRO, and VOCs were detected in soil samples collected from the sites, as presented in Table 10-1. Figure 10-1 provides an overview of the Peterson AFB OWS sites identified in the 2011 DERA investigation. Figures 10-2 and 10-3 show the DERA investigation sampling locations at Sites OW011 and OW012, respectively.

TABLE 10-1 2011 DERA Investigation Results

Detected Compounds	EPA Residential RSL ¹	Peterson AFB OW011	Peterson AFB OW012
Sample depth bgs	_	12–13 ft	8–9 ft
TPH-DRO	500*	2,400	400
TPH-GRO	500*	240	86
1,2,4-Trimethylbenzene	62	5	25
1,3,5-Trimethylbenzene	780	1.7	4.9
Benzene	1.1	_	0.088
Ethylbenzene	5.4	0.32	1.1
Isopropylbenzene (diisopropyl ether)	2,400	0.23	0.64
Xylene (total)	630	1.86	6.7
n-Propylbenzene	3,400	0.64	1.8
Naphthalene	3.6	15	45
p-Isopropylbenzene	_	0.62	2.8
sec-Butylbenzene	_	0.44	1.7
Tetrachloroethene	22	_	0.43
Toluene	5,000	0.085	2.2
Trichloroethene	0.91	_	0.18

Notes:

Concentrations are in milligrams per kilogram (mg/kg)

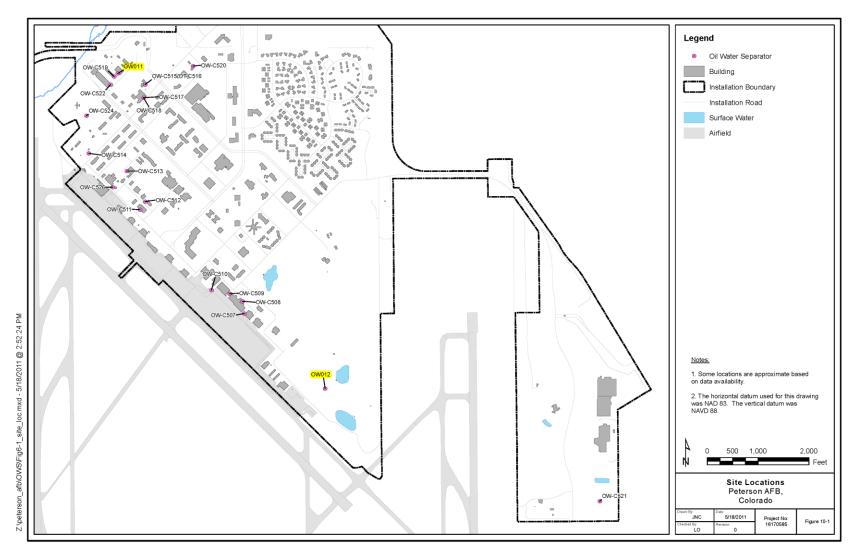
Results exceeding (U.S. Environmental Protection Agency Residential Regional Screening Levels) EPA RSLs are displayed in bold type

[&]quot;—" indicates non-detect values reported from the laboratory and/or no EPA RSL-R available.

^{*} Colorado Division of Oil and Public Safety Tier 1 risk-based screening level. Maximum detections are shown.

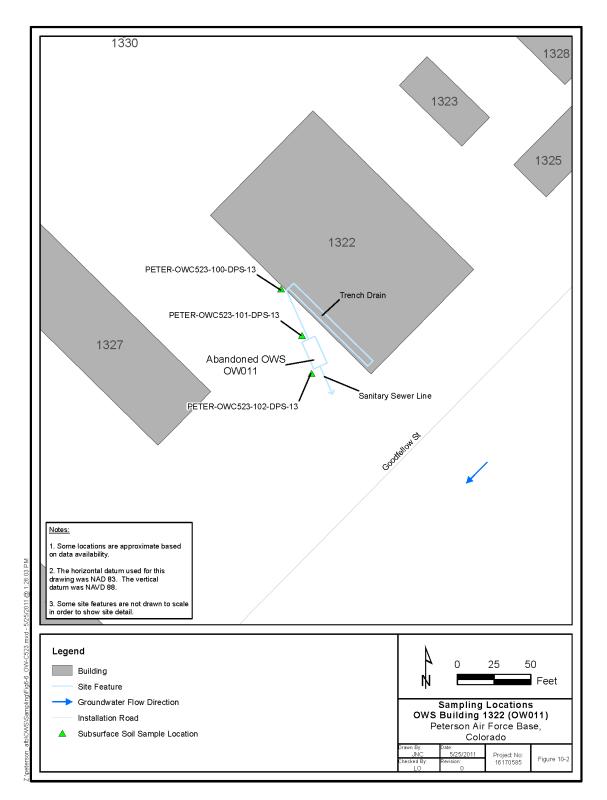
¹ EPA Residential RSLs, updated April 2012

Figure 10-1



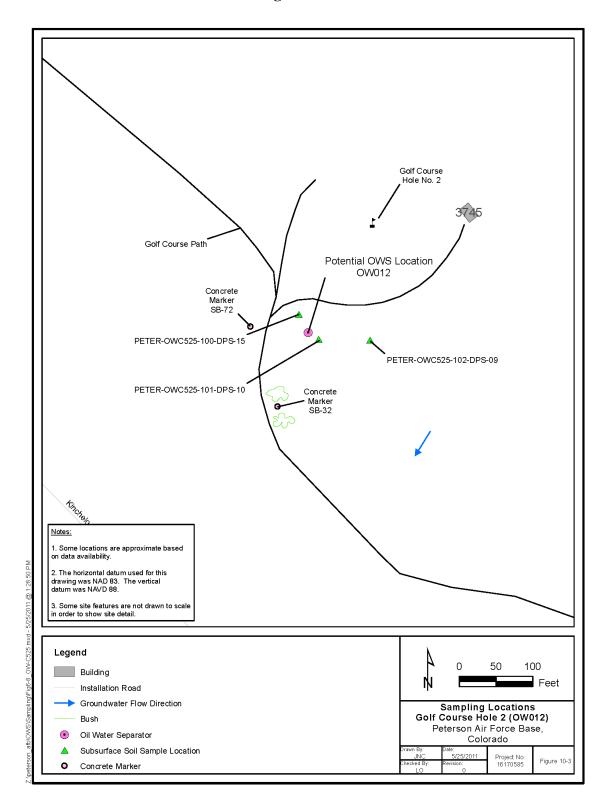
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Figure 10-2



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Figure 10-3



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10.3 Potential Impact to Groundwater

VOCs and TPH-DRO were detected in samples collected from Site OW011 during the 2011 DERA investigation. Detected compounds and the associated EPA RSL-Rs are presented in Table 10-1. Precipitation would have little effect on leaching contaminants further into the ground at Site OW011 because the OWS lies under a paved parking lot. Groundwater at Peterson AFBs is typically greater than 40 ft bgs; however, groundwater at site OW011 may be slightly shallower due to a nearby waterway. Because of the paved area and depth to groundwater, previous OWS operations are not anticipated to have any impact on the groundwater.

During the 2011 DERA investigation at Site OW012, one sample collected at approximately 8 to 9 ft bgs was analyzed. Naphthalene was detected at 45 mg/kg at 8-9 ft bgs, exceeding the EPA RSL-R of 3.6 mg/kg. Several other VOCs were detected but did not exceed EPA RSL-Rs. Groundwater is not anticipated to be affected at this location due to groundwater depths greater than 40 ft in the area. Soil samples will be collected to delineate soil contamination vertically and laterally at Site OW012 to confirm that that there is no impact to groundwater.

10.4 Baseline Risk Assessment

No previous risk assessments have been conducted at Site OW011.

Site OW012 is located within a former IRP site. A RI was conducted during 1989. Baseline humanhealth and ecological risk assessments were performed, as part of the RI, to evaluate if site-related chemicals were posing unacceptable risks to human health and the environment. Based on the data and information collected during the RI and the determination of no adverse impact to human health or the environment through risk assessment, no further action was required at the leach field site.

10.5 Land Use Considerations

Site OW011 is located adjacent to the building in the parking lot near the southwest wall of the Materials Supply Shop (Building 1322) in front of Bays 2 and 3. The current and foreseeable use of the property is for industrial activities, and groundwater is not used at Peterson AFB. However, the site will be evaluated for unrestricted residential land use.

The OW012 site includes an area near Hole 2 at the golf course. The current and foreseeable use of the property is for recreational activities, and groundwater is not used at Peterson AFB. However, the site will be evaluated for unrestricted residential land use.

10.6 Site Characteristics

10.6.1 Geology

Peterson AFB is situated on the southwestern flank of the Denver Basin, overlying steeply dipping Cretaceous bedrock. Quaternary alluvium blankets the northeastern dipping bedrock with coarse sand sediments up to 50 ft thick. The primary aquifers underlying the base are in the Quaternary alluvium and the Laramie-Fox Hills formation. The two main types of alluvium that occur on Peterson AFB are Broadway Alluvium and Piney Creek Alluvium. The Broadway Alluvium consists of poorly sorted, yellowish-brown coarse sand with high permeability. The more recent Piney Creek Alluvium occurs along the East Fork Sand Creek in the floodplain of the creek. The sediment is poorly sorted, gray to brown, humic-rich, firmly compacted clayey silt and sand up to 20 ft thick. The Piney Creek Alluvium is

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distinguishable from the Broadway Alluvium primarily by its greater clay and silt content and an associated low to medium permeability. The Laramie-Fox Hills sand unit outcrops on the northwest margin of Peterson AFB, along East Fork Sand Creek. The Laramie—a dark grayish-brown, iron-stained, fine-grained sand containing seams of lignite—reaches thickness of up to 250 ft. Thin sandstone beds in the lower part of Laramie yield moderate supplies of water. The Fox Hills Sandstone is a light olive-gray, thin-bedded, friable sandy shale occurring in the lower half of the sand unit. The lower sandstone beds are in the Laramie-Fox Hills aquifer (Sky Research Inc. 2010).

10.6.2 Hydrogeology and Hydrology

Underlying the surface hydrology of the area is a somewhat complicated system of bedrock and near-surface groundwater aquifers. Large quantities of groundwater are stored in the Dawson, Denver, Arapahoe, and Laramie-Fox Hills aquifers of the Denver Basin underlying much of northeastern to north-central El Paso County. Much of the water contained in the upper layers and outer boundaries of this formation is considered to be tributary to surface-water sources (URS, 2010). A number of monitoring wells have been completed on Peterson AFB, and water levels obtained from the wells indicate that depth to groundwater ranges from approximately 15 to 50 ft bgs along the northwest area of the installation. In the area of the golf course, the groundwater was found to be somewhat deeper, ranging between 43 and 55 ft bgs. In the southern end of the installation, no groundwater was found in the alluvium. This indicates groundwater is primarily associated with the areas near the creek and on the western edge of the installation and is deeper in the southeastern areas of the installation. The monitoring well data indicate that groundwater, where present, is generally flowing south-southeast across the installation (URS, 2011).

10.6.3 Nature and Extent of Contamination

The extent of contamination in soil was evaluated in the 2011 DERA investigation. Contaminants of Interest (COI) associated with Site OW011 were found at 12 to 13 ft bgs, and COIs associated with Site OW012 were found at 8 to 9 ft bgs. No groundwater samples were collected during this investigation.

10.7 Data Gaps and Uncertainties Associated with the Conceptual Site Model

Further investigation of Site OW011 and Site OW012 are needed as COIs were detected at concentrations above EPA RSL-Rs during the 2011 DERA investigation. Additional soil sample data are be required to confirm and further evaluate the COIs identified during the DERA investigation and to delineate and excavate soil contamination, if necessary. The DERA investigation data is limited to one soil sample from each site. The sample analyzed from Site OW011 was collected from the anticipated OWS influent line at 12 to 13 ft bgs. The sample from Site OW012 was collected at 8 to 9 ft bgs approximately 100 ft east of the potential OWS location. Additional soil samples may be collected at OW011 to evaluate the EPC for COIs. If EPC calculations are performed, a minimum of 10 non-biased soil samples will be collected from 0 to 12 ft bgs within 150 ft laterally of OW011. Because Site OW011 is paved, no surface soil samples (0 to 2 inches bgs) need to be collected. Site OW011 is in a paved parking lot and leaching of contaminants due to precipitation will be minimal.

Additional soil samples were collected near OW012 as part of the IRP site closure. Seven soil borings were drilled at the leach field and sampled continuously to 10 ft bgs and then at 5 ft intervals up to 71 ft bgs. Soil analytical data included 89 soil samples, which were analyzed using an on-site gas chromatograph (GC). Of those samples, 18 (along with 3 surface samples) were sent to an off-site laboratory for analysis. Samples from six monitoring wells installed as part of the IRP were also analyzed at an off-site lab. Sample results presented in the final RI report did not indicate any potential risk to human health or the environment.

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If field observations indicate the presence of contamination, non-biased soil samples from surface (0 to 2 inches bgs) and subsurface (0 to 12 ft bgs) intervals may be collected to support risk-based evaluation of any detected compounds, as appropriate. EPC calculations may be used to support risk-based evaluations. .

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QAPP Worksheet #11: Project/Data Quality Objectives (UFP-QAPP Manual Section 2.6.1) (EPA 2106-G-05 Section 2.2.6)

11.1 State the Problem

Available soil data is insufficient for delineating the COIs, determining potential impact to groundwater, and performing risk-based evaluation of each site, as appropriate. The data gaps in the Conceptual Site Model (CSM), identified in WS#10, must be addressed.

Site Characterization Site OW011:

- 1) The current data set for Site OW011 site consists of results from one sample collected in April 2011 from approximately 12 to 13 ft bgs at the anticipated OWS influent line. Analytical data for the soil sample indicated that TPH-DRO exceeds Colorado Division of Oil and Public Safety Tier 1 risk-based screening levels. VOCs were also detected in the soil sample. All VOC results were below the EPA RSL-R except for naphthalene which was detected at 15 mg/kg, compared to the EPA RSL-R of 3.6 mg/kg. Table 10-1 presents the detected compounds from Site OW011.
- 2) Due to the limited data from Site OW011, additional soil samples re-required to fully characterize the extent of potential contamination.
- 3) Additional samples will be collected from the potential leakage points of the OWS system, including trench drain effluent line, OWS influent line, and OWS effluent line. Biased soil samples will be collected from depths ranging from 5 to 15 ft bgs to determine the full extent of potential contamination, as needed, and to confirm if contamination exists. Soil samples will be analyzed for VOCs and SVOC, as presented in WS #15.
- 4) Biased soil sample results will be used to determine the lateral and/or vertical delineation of contamination, if encountered.
- 5) Non-biased soil samples may also be collected to support risk-based evaluation of the COIs.

Site Characterization Site OW012:

- 1) The current data set for Site OW012 consists of results from one sample collected in April 2011. This sample was collected approximately 100 ft east of the potential OWS location at 8 to 9 ft bgs. In 1988, samples were collected as part of a RI in the vicinity of Site OW012. WS #10 of this document discusses the results of the RI. The RI Decision Document eliminated the leach field from the IRP, and no further action was required.
- 2) During the 2011 DERA investigation, a sample collected from approximately 8 to 9 ft bgs from Site OW012 was analyzed for TPH-GRO, TPH-DRO, and VOCs. The soil sample results indicate that naphthalene exceed the associated EPA RSL-R. TPH-GRO, TPH-DRO, and several other VOCs were detected in the sample, and the results were below associated EPA RSL-Rs, as presented in Table 10-1.

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- 4) Additional soil samples will be collected from Site OW012 to confirm the naphthalene detection and to delineate the vertical and horizontal extent of the COIs that exceed the EPA RSL-Rs, as appropriate. Biased samples will be collected within the vicinity of samples collected during the 2011 DERA investigation. Figure 10-3 presents 2011 DERA investigation sampling locations. Soil sample data collected during this SI will be combined with data from previous investigations to fully characterize the site and revise the CSM. Samples will be analyzed for VOCs and SVOCs identified in WS #15. Field observations from biased samples will determine if further lateral and/or vertical delineation of contamination is required. If any visual staining or hydrocarbon odor is observed, or if PID readings indicate the presence of VOCs, additional stepout and step-down sampling will be completed.
- 5) Non-biased soil samples may be collected to support risk-based evaluation of the COIs.

11.2 Identify the Goal of the Study

The primary goal of the study is to collect additional and sufficient data to revise the CSM and determine whether further evaluation or remedial action is necessary to support unrestricted residential use.

11.3 Identify Information Inputs

Soil samples collected from Site OW011 and Site OW012 will be analyzed for VOCs and SVOCs as listed in WS #15. Soil samples will be analyzed using Solid Waste (SW)-846 Methods 8260B for VOCs and 8270C or D in Selective Ion Monitoring (SIM) mode for SVOCs. Additional soil data are required to eliminate data gaps in the CSM, to support unrestricted residential use, and to evaluate if remedial action is necessary to achieve unrestricted residential use of each site. Analytical sample results will be screened against EPA RSL-Rs for each COI to determine if further evaluation is needed. Further evaluation of the data will be used to revise the CSM and may include EPC calculations to determine if any COIs detected pose a risk to human health and the environment.

Data collected in previous investigations, identified in WS # 13 of this document, will be used to supplement data collected during this SI. Possible uses of previous data include, but may not be limited to, determining site boundaries, identifying COIs, and providing background on site geology and hydrogeology.

11.4 Define the boundaries of the Study

Site OW011 includes an abandoned OWS located in the parking lot of the southeast wall of the Materials Supply Shop, Building 1322, in front of Bays 2 and 3. The dimensions of the OWS are approximately 4 ft by 4 ft by 9 ft. Building 1322 represents a physical boundary for this SI. Samples are not anticipated to be collected beneath the building. The lateral and vertical site boundaries will be further defined based on the extent and concentration of contamination found during delineation sampling. Site boundaries will be created to exclude contamination discovered during delineation that is determined to be from another source.

Site OW012 includes an area near Hole 2 at the Peterson AFB golf course. The OWS was part of a former leach field system consisting of a settling tank and oil/water separator on a gravel-envelope drainage (leach) field. The lateral and vertical site boundary will be further defined based on the extent and concentration of contamination found during delineation sampling. Site boundaries will be created to exclude contamination that is determined to be from another source, if discovered during delineation.

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Soil samples collected from Site OW011 and OW012 will be analyzed for the VOCs presented in WS #15-1 and SVOCs presented in WS # 15-2. The analyte list was developed based on previous detections from the site and historic information on wastes that entered the OWS.

The temporal boundaries of the project are presented in WS # 14/16 and Figure 14-1, Project Schedule. There are no resource or schedule constraints for implementation of this UFP-QAPP at either site.

11.5 Develop the Analytical Approach

Soil samples will be analyzed using SW-846 Methods 8260B for VOCs and 8270C or D SIM for SVOCs. Results will be compared to the EPA RLR-Rs, which are the Project Action Limits (PALs), as presented in WS # 15. WS# 17 presents more detail on the sampling approach, including the proposed soil sample locations (Figures 17-1 and 17-2).

Site Characterization Site OW011 and OW012:

Biased and delineation soil samples will be collected from Sites OW011 and OW012, as identified in Figure 17-1 and Figure 17-2, respectively. Delineation sampling will continue as step-outs or step-downs until analytical results are below associated EPA RSL-Rs presented in WS# 15 or the site boundaries identified in section 11.4 of this WS are reached.

- **If** sample results from the proposed soil sample locations (Figures 17-1 and 17-2) are below the associated EPA RSL-Rs listed in WS #15, **then** biased sampling for COI delineation will cease.
- If field observations and/or elevated PID readings indicate the presence of COIs, then a sample will be collected for laboratory analysis and the step out step down process will continue.
- If field observations and/or PID readings indicate that no COIs are present during the step out/step down process, then a sample will be collected for laboratory analysis at that interval to verify PALs listed in WS# 15 have been achieved and one additional step out/step down interval sample will be collected and placed on hold at the lab pending results of the previous interval.
- If laboratory results exceed the PALs listed in WS# 15 from samples that were collected when field observations and PID readings indicated that no COIs were present, then the final step out/step down samples on hold at the laboratory will be analyzed to verify PALs have been achieved.

Delineation sampling will continue as step-outs or step-downs until analytical results are below associated EPA RSL-Rs presented in WS # 15 or the site boundaries identified in section 11.4 of this WS are reached.

Non-biased soil samples may be collected to support risk-based evaluation at each site, as appropriate.

- If a point-by-point comparisons of non-biased soil sample results show that all of the constituents detected are below the EPA RSL-R values listed in WS # 15, then an NFA determination will be pursued for that Site.
- If a point-by-point evaluation of non-biased soil samples shows results exceed the EPA RSL-Rs listed in WS # 15, then the derivation of an EPC for that area may be conducted.
- If the derived EPC shows contaminants pose a risk to human health or the environment, then the areas with results exceeding EPA RSL-Rs may be excavated or additional non-biased and or/biased samples may be collected.

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11.6 Specify Performance or Acceptance Criteria

The purpose of establishing decision-error tolerances is to set a limit (i.e., control) on the degree of uncertainty with which the decision is made and avoid, to the extent possible, the consequences of making an incorrect decision. To determine decision-error tolerances, potential sources of error must be identified and evaluated for the likelihood that an incorrect decision may result. Decision error can be minimized and controlled, but never totally eliminated. That is, decisions are made based on known, reliable, and reproducible data in which the opportunities for introducing unpredicted error are minimized to the degree possible. Establishing acceptable decision-error tolerances minimizes the three types of error listed below:

- **Sampling design error** occurs when the sampling design does not account for the natural variability in the true state of the environment (i.e., does not produce representative data).
- **Measurement error** occurs as a result of random and systematic errors that are inherent in the each step of the data production process, including sample collection, preparation and analysis, and data reduction, handling, and reporting.
- Total study error is a function of both sampling design and measurement error combined.

The objectives of establishing error tolerances are to create limits for which data can be used, which will minimize the opportunity for introducing manageable error in the decision-making process; and to limit the consequences of implementing an incorrect decision. The consequences of implementing a full-scale remedial design, based on data for which decision errors were not developed, potentially could result in a failure of the remedial action to achieve site closure based on EPA RSL-R values.

- Measurement Quality Objectives (MQOs) are project-specific, analytical parameters derived from project-specific Data Quality Objective (DQO)s. MQOs include the QA activities that will be conducted during the project, and QC acceptance criteria for the data quality indicators (DQIs). MQOs establish the minimum for analytical performance parameters (i.e., serve to specify how good data must be) derived from the level of performance needed to achieve the project goals (as expressed in the DQOs). Project MQOs are not intended to be technology- or method-specific, and generally will not specify the methods by which the data are generated. MQOs consist of QA activities (i.e., calibration, data assessment and reporting, preventive maintenance, and corrective action), DQIs, and QC acceptance criteria. MQOs are presented in WSs #12 and #28.
- Measurement Performance Criteria (MPC) may be general or specific criteria (such as QC method acceptance limits) that are used by a project to judge whether a laboratory can perform a specified activity to the defined standard. Establishing QC acceptance criteria for the MPCs sets quantitative goals for the quality of data generated in the analytical measurement process or measurement systems (EPA 1998). MPC for this project are based upon DoD Quality Systems Manual (QSM) limits and will be reported in laboratory data packages. MPC are presented in WS #12 and WS #28.

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• QC Acceptance Criteria are method- and technology-specific protocols and specifications that demonstrate that data of known and sufficient quality are generated. QC acceptance criteria include specific limits for sensitivity, recovery, memory effects, limit of quantitation, repeatability, and reproducibility, and are designed such that if they are consistently met, the project MQOs will be achieved, and the resulting data will be sufficient to meet the project DQOs and support the project decisions (Crumbling 2001).

Laboratory DLs, Limit of Detection, and LOQs were compared to PALs and determined to be usable for project purposes; relevant details are presented in WS #15. Applicable DoD QSM criteria were assessed and determined to be acceptable for controlling bias; relevant details are presented in WS #12 and WS #28. If a data validator applies professional judgment to determine any of the described criteria do not apply, reasoning will be clearly and concisely documented in the data validation reports.

11.7 Develop the Plan for Obtaining Data

Soil samples will be collected using direct-push technology (DPT) as described in section WS #17. Surface soil samples at Site OW012 may be collected using a hand auger, shovel, or equivalent in lieu of the DPT, if appropriate. Anticipated sample locations are presented in Figure 17-1 for Site OW011 and Figure 17-2 for Site OW012. Soil samples will be analyzed by SW-846 8260 and 8270 SIM for compounds identified in WS #15. Based on the low levels detections at Site OW012 from previous investigations and the anticipated depth to groundwater exceeding 40 ft bgs, no groundwater samples are planned for this SI.

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QAPP Worksheet #12: Measurement Performance Criteria (UFP-QAPP Manual Section 2.6.2) (EPA 2106-G-05 Section 2.2.6)

MPC established to ensure data collected satisfy project-specific DQOs are defined in the subsequent worksheets. Data will be qualified as described in WS #12 and WS #28. If professional judgment is used to apply/not apply qualifiers as described, the reasoning will be clearly and concisely documented in the relevant data validation report.

Matrix: Soil

Analytical Group or Method: VOC/ SVOCs

Concentration Level: Low

Data Quality Indicator	QC Sample or Measurement Performance Activity	Measurement Performance Criteria
		Relative percent difference (RPD) $\leq 50\%$ when target elements are detected
Overall Precision	Field Duplicates	in both samples ≥ sample-specific Level of Quantitation (LOQ)
		Qualify affected sample results J/UJ for exceedances
Analytical Precision	Laboratory Control Sample Duplicates	RPD ≤ 30%
(laboratory)	Laboratory Control Sample Duplicates	Qualify affected sample results J/UJ for exceedances
		DoD QSM Limits
Analytical Accuracy/Bias	Laboratory Control Samples	Qualify affected detections J for recoveries above limits
(laboratory)		Qualify affected results J/UJ for recoveries below limits
		Reject affected nondetect results for recoveries < 10%
		DoD QSM Limits
Analytical Accuracy/Bias	Matrix Spike Duplicates	Qualify affected detections J for recoveries above limits
(matrix interference)	Wattix Spike Duplicates	Qualify affected results J/UJ for recoveries below limits
		Reject affected nondetect results for recoveries < 10%
Overall Accuracy/Bias	Equipment Blanks	No target analyte concentrations $\geq 1/2$ LOQ or qualify as described for
(contamination)	Equipment Blanks	Method Blanks in WS # 28.
		Recovery within ±25% of LOQ. Assess direction of bias for associated
Sensitivity	LOQ Verification Sample (spiked at LOQ)	outlier QC or calibration results. For impact on usability of data for project
		purposes, use professional judgment.
Completeness	See WS #34	See WS #34

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QAPP Worksheet #13: Secondary Data Uses and Limitations (UFP-QAPP Manual Section 2.7)

(EPA 2106-G-05 Chapter 3: QAPP Elements for Evaluating Existing Data)

Data type	Source	Data uses relative to current project	Factors affecting the reliability of data and limitations on data use
Concentration of VOCs	Final Technical Evaluation	Provides sample results and site	Analytical sample results are limited to
in soil, site-specific	Report. Investigation of OWSs	condition information for remedial	one depth interval and one sample from
hydrology, lithology, and	to Determine DERA Eligibility,	approach	each site
geology.	July 2011		
Concentration of	U.S. Air Force Installation	Provides sample results and site	Data are based on 1989 sampling and
contaminants in soil,	Remediation Program, Remedial	condition information for remedial	analysis.
site-specific hydrology,	Investigation/Feasibility Study at	approach at Site OW012	
lithology, geology,	Peterson AFB, Colorado		
boring logs, and risk-	Springs, CO September 1989		
assessment data.			

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QAPP Worksheet #14/16: Project Tasks and Schedule (UFP-QAPP Manual Section 2.8.2) (EPA 2106-G-05 Section 2.2.4)

Data Management Tasks

All data will be managed using a central environmental database/environmental data management system (EDMS) for data management. This database is a secure repository for all project data including chemical analyses, geophysical, and Global Information System data. Data is protected with nightly full and incremental data backups to online drive array, and tape. Three months of data is stored at any one time. A weekly backup is stored off-site for safekeeping and disaster recovery purposes. For chemical analyses, laboratory electronic data deliverable (EDD)s will be submitted directly to the database and an immediate compliance check is performed, which will help prevent errors between field and lab data.

Automated data review will be performed using the EDMS, with corollary checks of calibration, sample receipt, and field data by qualified data validators. Data will be validated in accordance with project-specific guidelines as identified in this document.

The database will provide a direct translation of all data types to the ERPIMS format as an end deliverable. All project documents and data will be stored by RMA-Insight for 5 years after the period of performance ends on July 15, 2020.

Documentation and Records

Information documented in dedicated field logbooks will include some or all of the following:

- Date and time of entry onto site
- Names of personnel on site
- Number of samples taken
- Sample collection methods
- Description of sampling points
- Date and time of collection
- Sample identification numbers
- Sample start and finish times
- Site temperature and atmospheric pressure
- Summary of field task related to sampling
- Decontamination procedures
- Records of telephone conversations
- Calibration of equipment used
- Field Corrective Action (CAs) taken
- Record of any damage to property along with a description of repairs

Field sampling personnel will properly identify all samples taken in the field with an adhesive sample label attached to each sample container. The sample label will contain the site name, field identification number, date, time, location of the sample collected, and identification of preservatives used. Sample information will be legibly printed with waterproof ink. The sample identification numbers will be used

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on field sheets, Chain-of-Custody (COC) forms, and other documentation records. Information documented will be provided to AFCEC for administrative records.

COC forms will be used to document the integrity of all samples collected. To maintain a record of sample collection, transfer between personnel, shipment, and receipt by the laboratory, COC forms will be filled out for sample sets as determined appropriate during the course of fieldwork (SOP No. 3 – Sample Handling and Management). All samples will be shipped via Federal Express priority overnight or hand delivered to the laboratory the same day as sample collection.

Data Packages

All fixed laboratory analytical data will be provided in Contract Laboratory Program-Like or Level-IV data packs with all relevant summary forms, printouts, and raw instrument data to enable full third-party data validation.

Assessment/Audit Tasks

No external laboratory audits are planned for this project.

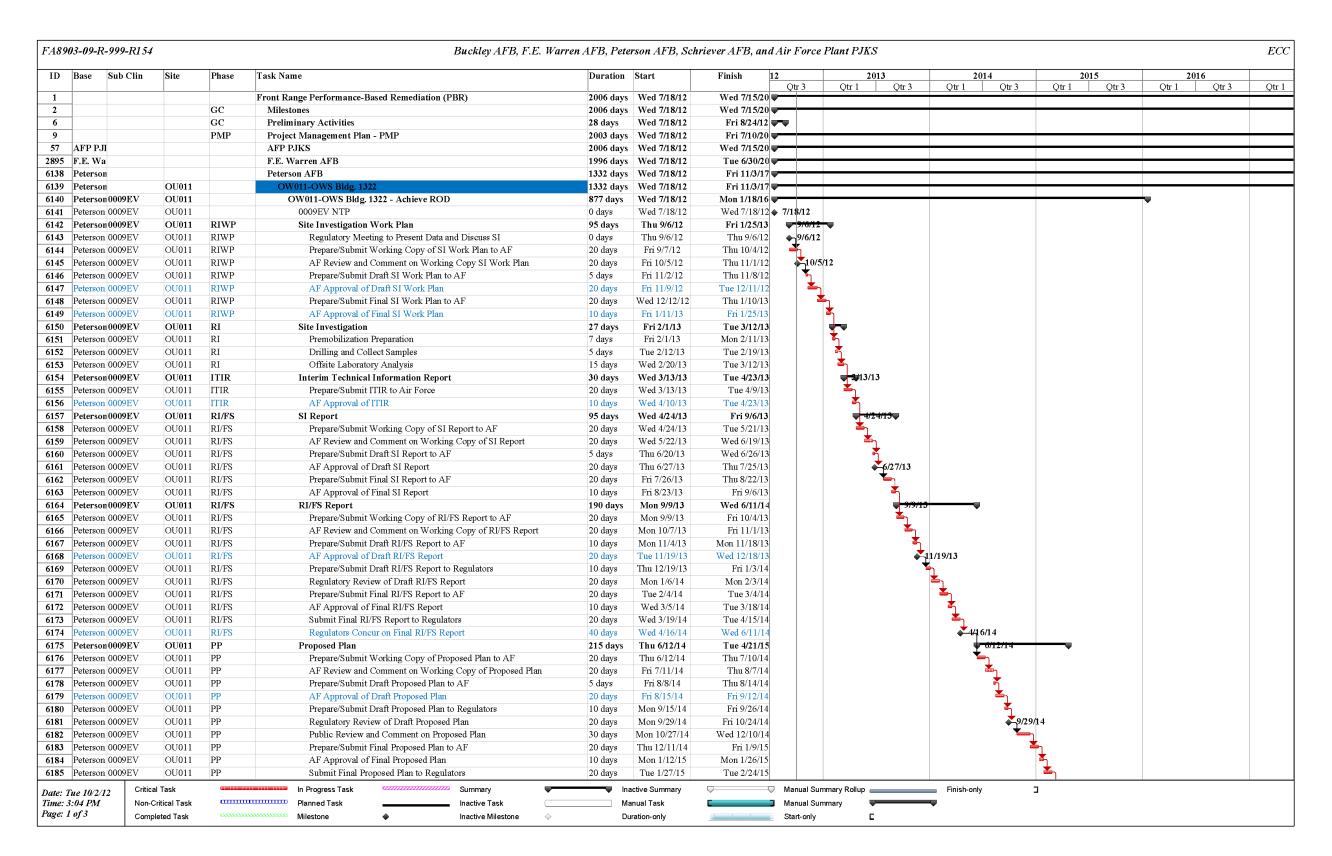
Data Review Tasks

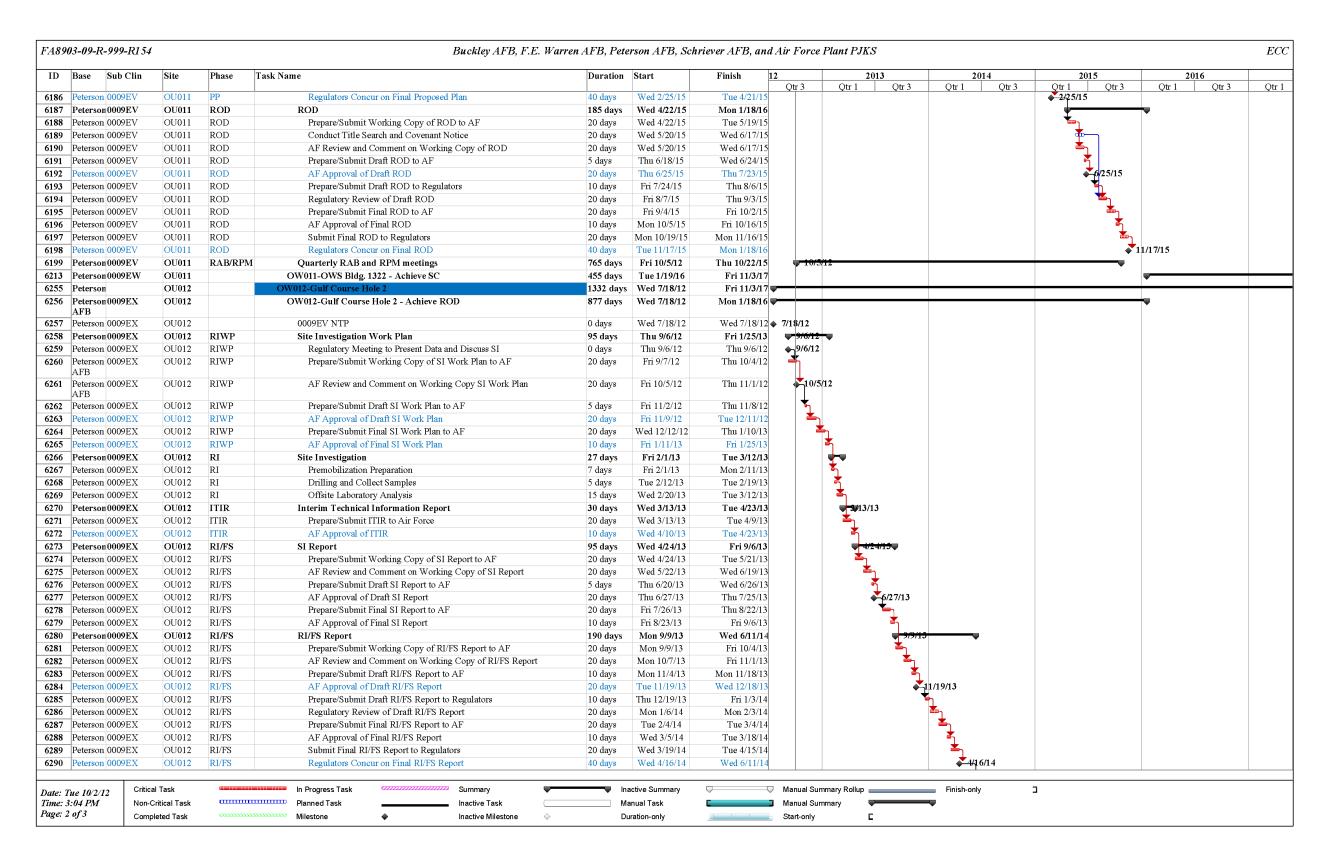
Prior to releasing the data packages, TA will review all analytical data in order to verify that:

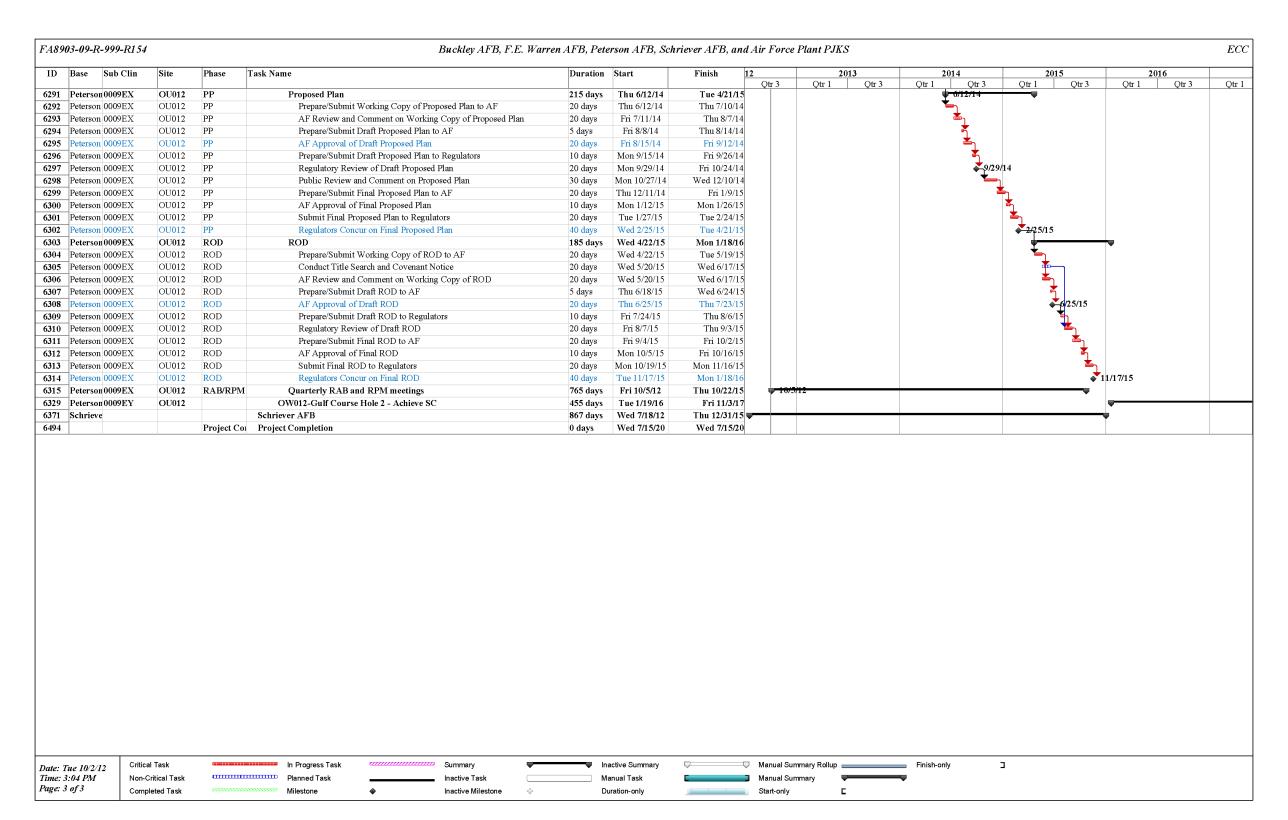
- All analytical results are correct and complete
- The appropriate SOPs have been followed and are identified in the project records
- Proper documentation procedures have been followed
- Any non-conformances have been documented

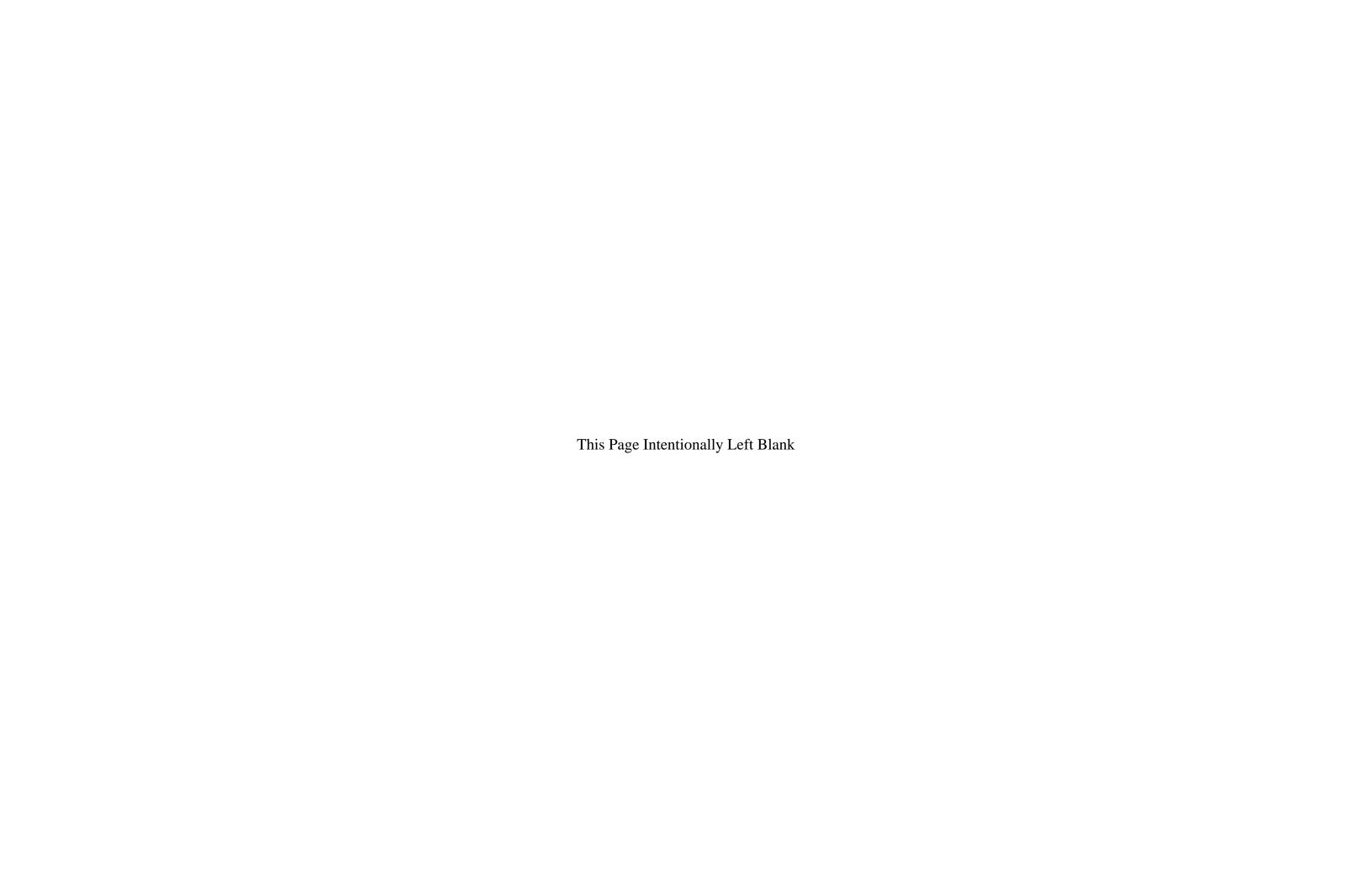
The project schedule is shown on the following pages.











QAPP Worksheet #15-1: Project Action Limits and Laboratory-Specific Detection/Quantitation Limits, VOCs (UFP-QAPP Manual Section 2.6.2.3 and Figure 15) (EPA 2106-G-05 Section 2.2.6)

Matrix: Soil

Analytical Method: 8260B Concentration Level: Low

Analyte ¹	Project Action Limit (PAL)	PAL Reference ²	Project Quantitation Limit Goal	Laboratory- Specific Quantitation Limit ³	Laboratory- Specific Detection Limit ⁴
1,2,4-Trimethylbenzene	62,000	EDA DOL D	(μg/Kg) 31,000	(μg/Kg) 5.0	(μg/Kg) 0.58
·		EPA RSL-R			
1,3,5-Trimethylbenzene	780,000	EPA RSL-R	390,000	5.0	0.57
2-Butanone (MEK)	28,000,000	EPA RSL-R	14,000,000	20	1.83
4-Methyl-2-pentanone (MIBK)	5,300,000	EPA RSL-R	2,650,000	20	4.36
Acetone	61,000,000	EPA RSL-R	30,500,000	20	5.38
Benzene	1,100	EPA RSL-R	550	5.0	0.47
Ethylbenzene	5,400	EPA RSL-R	2,700	5.0	0.67
Isopropylbenzene / (diisopropylether)	2,400,000	EPA RSL-R	1,200,000	5.0	0.59
m-Xylene & p-Xylene	1,190,000	EPA RSL-R	595,000	3.2	1.04
Naphthalene	3,600	EPA RSL-R	1,800	5.0	0.63
n-Butylbenzene	3,900,000	EPA RSL-R	1,950,000	5.0	0.56
n-Propylbenzene	3,400,000	EPA RSL-R	1,700,000	5.0	0.58
o-Xylene	690,000	EPA RSL-R	345,000	5.0	0.61
Styrene	6,300,000	EPA RSL-R	3,150,000	5.0	0.63

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Analyte ¹	Project Action Limit (PAL)	PAL Reference ²	Project Quantitation Limit Goal	Laboratory- Specific Quantitation Limit ³	Laboratory- Specific Detection Limit ⁴
	(µg/Kg)		$(\mu g/Kg)$	$(\mu g/Kg)$	$(\mu g/Kg)$
Tetrachloroethene	22,000	EPA RSL-R	11,000	5.0	0.59
Toluene	5,000,000	EPA RSL-R	2,500,000	5.0	0.69
Trichloroethene	910	EPA RSL-R	455	5.0	0.23

¹ Non-detects will be reported down to the detection limit (DL) in support of PALs. In the event that current technology does not support the PAL, a non-detect result reported to the DL will be considered acceptable.

² EPA residential screening level for residential soil (RSL-R), updated April 2012

³ Lowest calibration concentration.

 $^{^4}$ Statistically derived value; the lowest concentration detectable by the laboratory with 99% confidence analyte is present. $\mu g/Kg$ – micrograms per kilograms

QAPP Worksheet #15-2: Project Action Limits and Laboratory-Specific Detection/Quantitation Limits, SVOCs

Matrix: Soil

Analytical Method: 8270C/D SIM PAH

Concentration Level: Low

Analyte ¹	Project Action Limit (PAL) ² (µg/Kg)	PAL Reference	Project Quantitation Limit Goal (µg/Kg)	Laboratory- Specific Quantitation Limit ³ (µg/Kg)	Laboratory- Specific Detection Limit ⁴ (µg/Kg)
Acenaphthene	3,400,000	EPA RSL-R	1,700,000	5	0.16
Anthracene	17,000,000	EPA RSL-R	8,500,000	5	0.72
Benzo[a]anthracene	150	EPA RSL-R	75	5	0.90
Benzo[a]pyrene	15	EPA RSL-R	7.5	5	0.74
Benzo[b]fluoranthene	150	EPA RSL-R	75	5	1.2
Benzo[k]fluoranthene	1,500	EPA RSL-R	750	5	1.0
Chrysene	15,000	EPA RSL-R	7,500	5	1.0
Dibenz(a,h)anthracene	15	EPA RSL-R	7.5	5	1.3
Fluoranthene	2,300,000	EPA RSL-R	1,150,000	5	1.0
Fluorene	2,300,000	EPA RSL-R	1,150,000	5	0.47
Indeno[1,2,3-cd]pyrene	150	EPA RSL-R	75	5	1.1
Pyrene	1,700,000	EPA RSL-R	850,000	5	1.1

¹ Non-detects will be reported down to the detection limit (DL) in support of PALs. In the event that current technology does not support the PAL, a non-detect result reported to the DL will be considered acceptable.

² EPA residential screening level for residential soil (RSL-R), updated April 2012

³ Lowest calibration concentration.

⁴ Statistically derived value; the lowest concentration detectable by the laboratory with 99% confidence analyte is present.

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QAPP Worksheet #17: Sampling Design and Rationale (UFP-QAPP Manual Section 3.1.1) (EPA 2106-G-05 Section 2.3.1)

This worksheet describes the sampling design for the SI at Sites OW011 and OW012 at Peterson AFB and the basis for its selection. The sampling design is judgmental and focuses on refining the CSM by confirming previous analytical results, defining the potential extent of soil contamination, and completing a risk-based evaluation of COIs, as appropriate. Data must be sufficient to either support closure of a site based upon unrestricted residential land use or development of a remedial action to achieve such a closure. Biased soil samples will be collected where COIs were previously detected or may be encountered based upon known site history. Judgmental and biased soil sampling based on previous results and field observations will ensure that the full extent of contamination at Sites OW-011 and OW012 is identified.

At Site OW011, soil sample locations include the approximate location of the former trench-drain effluent line, OWS influent line, and OWS effluent line. At Site OW012, biased soil sample locations are proposed within the vicinity of previous detections identified during the 2011 DERA investigation and at the approximate OW012 influent and effluent lines. Biased samples will be collected and analyzed to determine the existence of VOC and SVOC soil contamination, if present. Samples at these sites will not be analyzed for metals or PCBs because these constituents have not been detected in previous investigations. Additional step-out or step-down delineation samples may be collected laterally and vertically from locations of expected contamination. SOPs on sample collection procedures and handling, equipment decontamination, and handling of Investigation Derived Waste are referenced in WS # 21.

17.1 Site OW011 Sampling Approach

A DPT drill rig will be used to advance probes into the soil to collect soil samples at Site OW011 in the parking lot near the southwest wall of the Materials Supply Shop (Building 1322). Soil samples will be collected from the same vicinity as the 2011 DERA investigation to target the potential leakage points of the OWS. The trench-drain effluent line, OWS influent line, and OWS effluent line locations will be targeted for biased soil sampling (Figure 17-1). Soil samples will be collected continuously from 5 ft to 15 ft bgs. All soil borings will be screened with a PID. PID readings will be documented in the field logbook and used for informational purposes of determining step out/step down locations. Soil encountered with field observations of contamination, including elevated PID readings, visual staining, and hydrocarbon odor will be sent to an off-site laboratory for analysis. When an area of suspected contamination is encountered, a systematic approach of stepping down vertically no more than 2 ft and stepping out laterally no more than 5 ft will commence until field observations (including field instrument readings) indicate there is no contamination present in the step out location. Once field observations indicate the absence of contamination, samples will be collected at the location either adjacent to or below the previously observed contamination and placed on hold at the lab. Samples that are placed on hold at the laboratory will be analyzed if results in a preceding interval exceed EPA RSL-Rs. Laboratory data is considered critical and will be used to evaluate any risk-based closure or potential remedial action.

Random, non-biased samples may be collected to support risk-based evaluation of the site. The non-biased samples will be collected within a 150-ft-by-150-ft grid from depths up to 12 ft bgs. Rationale for sampling locations and depths will follow EPA Region 8 EPC guidelines.

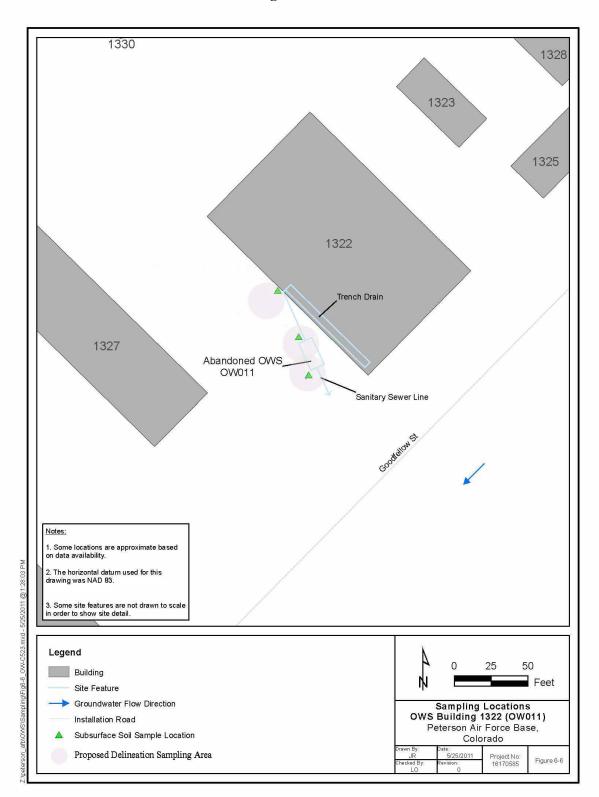
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TABLE 17-1 Site OW011 Sampling

Data Quality Objective	Potential Impact to Remedial Action	Data Collection Method	Analytical Methods
Determine if VOC or SVOC concentrations near the OWS near Building 1322 exceed EPA RSL-R values.	Extent of contamination will determine step-out and step-down delineation sampling points.	Collect samples using DPT drill rig at depths ranging from 5 ft to 15 ft bgs. (Sample locations are provided on Figure 17-1.)	8260 VOC and 8270 SIM SVOC
Derive EPC for VOC and SVOCs.	Evaluate the soil contamination over a wider area to determine if the level of contamination in an area represents an unacceptable threat to human health.	Collect samples using DPT drill rig over a 0.5-acre area. Evaluate 2 intervals: 0–3 inches and 0–12 ft bgs. The 0-3 inches bgs will not be collected at Site OW011 due to concrete and asphalt covering the site.	8260 VOC and 8270 SIM SVOC

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Figure 17-1



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17.2 Site OW012 Sampling:

A DPT drill rig will be used to advance probes into the soil to collect soil samples at Site OW012. Figure 17-2 presents locations where biased soil samples will be collected and approximate delineation sampling locations. Biased soil samples will be collected from the same vicinity where detections exceeded EPA RSL-R values during the 2011 DERA investigation and from the area near the estimated influent and effluent line connection points of the OWS. These three locations are representative of areas of known contamination and will be the starting points for a complete delineation of the site. Results from the biased samples will determine if and where any additional delineation sampling will occur as described in WS #11 Section 11.5 of this document. Soil samples will be collected continuously from 5 ft to 15 ft bgs or until refusal. Soil borings will be screened with a PID. PID readings will be documented in the field logbook and used for informational purposes of determining step out/step down locations. Soil encountered with elevated PID readings, visual staining, and hydrocarbon odor will be sent to an off-site laboratory for analysis. When an area of suspected contamination is encountered, a systematic approach of stepping down vertically no more than 2 ft and stepping out laterally no more than 5 ft will commence until field observations indicate there is no contamination present in the step out location or the DPT probes reach refusal, whichever occurs first. Once field observations indicate the absence of contamination, samples will be collected at the location either adjacent to or below the previously observed contamination and placed on hold at the laboratory. Samples that are placed on hold at the laboratory will be analyzed if results in a preceding interval exceed EPA RSL-Rs. Laboratory data is considered critical and will be used to evaluate any risk-based closure or potential remedial action.

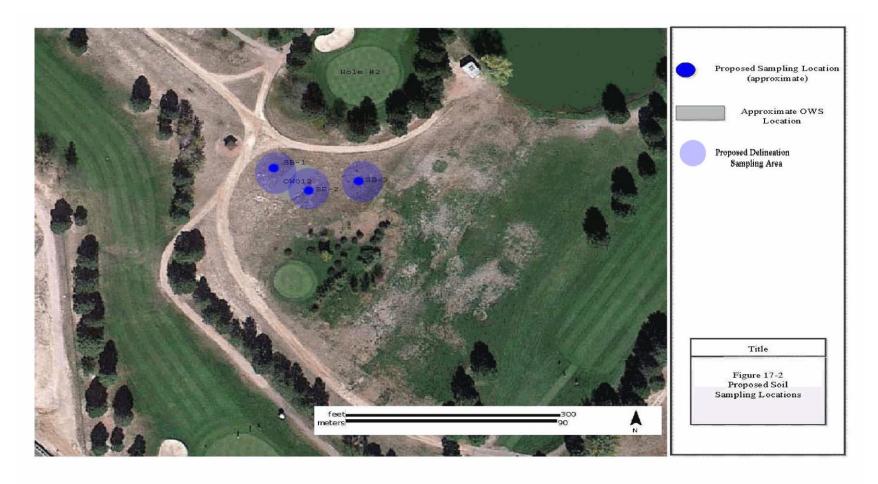
Sample depths will be located between 0 ft and 15 ft bgs. Soil samples from multiple intervals will be collected from each boring. Results from the biased samples will determine any delineation sampling laterally and vertically is required. Random, non-biased samples may be collected for EPC calculations. The non-biased samples will be collected within a 150-ft-by-150-ft grid from depths up to 12 ft.

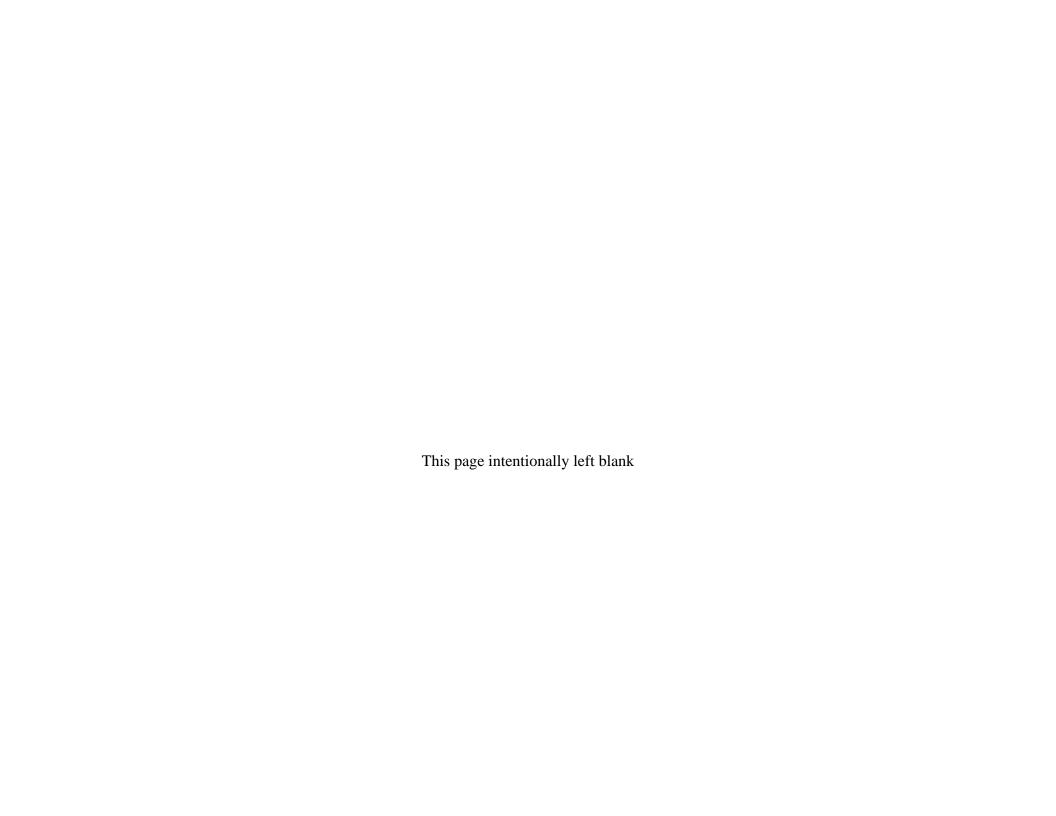
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TABLE 17-2 Site OW012 Sampling

Data Quality Objective	Potential Impact to Remedial Action	Data Collection Method	Analytical Methods
Determine if VOC or SVOC concentrations near the OWS site near Golf Course Hole #2 exceed EPA RSL-R values.	Extent of contamination will determine step-out and step-down sampling points for delineation.	Advance direct-push tooling to sample depths ranging from 5 ft to 15 ft bgs and collect soil samples. (Sample locations are provided on Figure 17-2.)	8260 VOC and 8270 SIM SVOC
Derive EPC for VOC and SVOCs	Evaluate the soil contamination over a wider area to determine if the level of contamination in an area represents an unacceptable threat to human health.	Collect samples using DPT drill rig over a 0.5-acre area. Evaluate 2 intervals: 0–2 inches and 0–12 ft.	8260 VOC and 8270 SIM SVOC

Figure 17-2





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QAPP Worksheet #18: Sampling Locations and Methods (UFP-QAPP Manual Section 3.1.1 and 3.1.2) (EPA 2106-G-05 Section 2.3.1 and 2.3.2)

Sample Identification	Matrix	Matrix Depth ² (ft bgs) (approximate) Type Analy		Analytical Group	Sampling SOP	Comments						
OW011 – Soil Samples												
PAFB-OW011-SB01-xx ¹	Soil	5- 15	Grab	VOCs/SVOCs	See WS #21							
PAFB-OW011-SB02- xx ¹	Soil	5-15	Grab	VOCs/SVOCs	See WS #21							
PAFB-OW011-SB03- xx ¹	Soil	5-15	Grab	VOCs/SVOCs	See WS #21							
,		0	W012 – Soil Sample	es								
PAFB-OW012-SB01- xx ¹	Soil	0-15	Grab	VOCs/SVOCs	See WS #21							
PAFB-OW012-SB02- xx ¹	Soil	0-15	Grab	Grab VOCs/SVOCs								
PAFB-OW012-SB03- xx ¹	Soil	0-15	Grab	Grab VOCs/SVOCs								

Samples presented in this table represent initial biased sample locations. Step-out/step-down sample locations and non-biased sample locations will follow the same sample naming convention.

 $^{^{1}}$ Sample identification will consist of soil boring (SB) numbers followed by the sample depth (xx)

² Sample collection depth will be determined based field observations including visual staining, hydrocarbon odor, and elevated PID readings.

QAPP Worksheet #19 & 30: Sample Containers, Preservation, and Hold Times (UFP-QAPP Manual Section 3.1.2.2) (EPA 2106-G-05 Section 2.3.2)

Laboratory:

TestAmerica-Denver, 4955 Yarrow Street, Arvada, CO 80002

Deb Henderer or To Be Determined Lab PM; <u>Debra.Henderer@testamericainc.com</u>; 303-736-0134

Required accreditations/certifications: DoD Environmental Laboratory Accreditation Program (ELAP) certification/Laboratory must be compliant with the current version of the DoD QSM

Backup Laboratory:

ALS Environmental, 225 Commerce Drive, Fort Collins, CO 80524 Amy Wolf; Amy.Wolf@ALSGlobal.com; 970-490-1511 extension 201

Sample Delivery Method: Federal Express; Hand Delivery

Analyte/ Analyte Group	Matrix	Method/ SOP	Accreditation Expiration Date	Container(s) (number, size & type per sample)	Preservation	Preparation Holding Time	Analytical Holding Time	Data Package Turnaround
Semi-volatile Organic Compounds	Soil	8270 SIM / DV-MS- 0002	31 Oct 2013	1, 4 oz, glass jar	4 ± 2°C	14 days	40 days	28 days
Volatile Organic Compounds	Soil	8260 / DV- MS-0010	31 Oct 2013	2, 25 g EnCore TM	4 ± 2°C	14/7 days— Preserved/ Unpreserved	14/7 days— Preserved/ Unpreserved	28 days

[°]C - degrees Centigrade

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QAPP Worksheet #20: Field QC Summary (UFP-QAPP Section 3.1.1 and 3.1.2) (EPA 2106-G-05 Section 2.3.5)

Matrix	Analyte/Analytical Group	Field Samples (estimated)	Field Duplicates	Matrix Spikes	Matrix Spike Duplicates	Field Blanks	Equipment Blanks	Trip Blanks	Other	Total # analyses
Soil	VOCs	25	1/10	1/OWS Site	1/OWS Site	0	2	5	N/A	37
Soil	SVOCs	25	1/10	1/OWS Site	1/OWS Site	0	2	NA	N/A	32

¹ Equipment rinsates will be collected for any parameters sampled using reusable equipment at a rate of 1/event. If disposable equipment is used, no equipment blanks will be collected. Equipment rinsates will consist of clean water run over or through decontaminated field sampling equipment.

² Trip blanks will be collected a rate of one/cooler containing samples for VOC analysis. Trip blanks should consist of 2-3 vials, as provided by laboratory, carried into the field and subjected to the same conditions as site samples. Trip blank vials will not be opened prior to analysis.

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QAPP Worksheet #21: Field SOPs (UFP-QAPP Manual Section 3.1.2) (EPA 2106-G-05 Section 2.3.2)

SOP # or reference	Title, Revision, Date, and URL (if available)	Originating Organization	SOP option or Equipment Type (if SOP provides different options)	Modified for Project? Y/N	Comments
SOP No. 1	Water Level Measurement (Appendix B)	RMA-Insight	Water Level Probe	N	
SOP No. 3	Sample Handling and Management (Appendix B)	RMA-Insight	N/A	N	
SOP No. 4 Sampling Equipment Decontamination (Appendix B)		RMA-Insight	Decontamination of sampling equipment, field monitoring equipment, and personnel	N	
SOP No. 5	P No. 5 Soil Sampling for Chemical Analysis (Appendix B) RMA-Insig		Stainless-steel trowel, shovel, scapula, coring device, trier, hand auger, or other appropriate hand tool Stainless-steel, split-spoon samplers Drilling rig or soil-coring rig Stainless-steel pan or bowl	N	
SOP No. 6	Investigation-Derived Waste Management (Appendix B)	RMA-Insight	N/A	N	
SOP No. 7	Lithologic Logging (Appendix B)	RMA-Insight	Munsell Soil Color Chart Soil Boring Log Form	N	

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QAPP Worksheet #22: Field Equipment Calibration, Maintenance, Testing, and Inspection (UFP-QAPP Manual Section 3.1.2.4) (EPA 2106-G-05 Section 2.3.6)

Field Equipment	Activity	SOP Reference	Title or position of responsible person	Frequency	Acceptance Criteria	Corrective Action
PID	Standard 2-point calibration (zero and span)	MiniRAE Manufacturer's Instructions Manual Rev. A April 2007 (Appendix B)	ECC PM or Designee	2 point calibration daily before use and between each biased sampling location.	Calibration successful	Clean filter and/or recalibration

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QAPP Worksheet #23: Analytical SOP's (UFP-QAPP Manual Section 3.2.1) (EPA 2106-G-05 Section 2.3.4)

SOP#	Title, Date, and URL (if available)	Definitive or Screening Data	Matrix/Analytical Group	SOP Option or Equipment Type	*Modified for Project? Y/N
DV- MS- 0010	Determination of Volatile Organics by GC/MS (SW846 8260B and EPA 624) Revision 7, 07/27/2012	Definitive	Soil/Volatiles	GC/MS	No
DV- MS- 0011	GC/MS Analysis Based on Method 8270C SIM and 625 Revision 6, 01/09/2012	Definitive	Soil/Semi-volatiles	GC/MS	No
DV- MS- 0012	GC/MS Analysis Based on Method 8270D SIM Revision 1.1, 12/01/2011	Definitive	Water & Soil/ Semi- volatiles	GC/MS	No
DV- OP- 0010	Soxhlet Extraction of Solid Samples (SW-846 3540C) Revision 5, 07/13/2012	Preparation	Soil/Organic Prep	NA	No
DV- OP- 0007	Concentration and Clean-up of Organic Extracts (SW-846 3510C, 3520C, 3540C, 3546, 3550B, 3550C, 3620C, 3660B, 3665A, and EPA 600 series) Revision 6, 10/14/2011	Preparation	Soil/Organic Prep	NA	No

[‡] A brief summary of project-specific SOP modifications must be provided on this worksheet or referenced.

Notes:

GC/MS - Gas Chromatography/Mass Spectrometry

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QAPP Worksheet #24: Analytical Instrument Calibration (UFP-QAPP Manual Section 3.2.2)

(EPA 2106-G-05 Section 2.3.6)

Instrument	Calibration Procedure	Calibration Range	Frequency	Acceptance Criteria	Corrective Action	Title/Position Responsible for Corrective Action	SOP Reference
GC/MS – 8270C/D SIM and 8260B	Tuning	See WS #15 for PAL/DL	Prior to initial calibration or continuing calibration verification, every 12 hours	Refer to criteria listed in the method SOP for Tune criteria	Retune the instrument and verify (instrument maintenance may be needed).	Lab Manager/ Analyst	DV-MS- 0010 & DV-MS- 0011
GC/MS – 8270C/D SIM	Breakdown check		At the beginning of each 12-hour period prior to analysis of samples	Degradation < 20% for dichlorodiphenyltrichloroethane (DDT). Benzidine and pentachlorophenol should be present at their normal responses, and should not exceed a tailing factor of 2.	Correct problem; then repeat breakdown check.	Lab Manager/ Analyst	DV-MS- 0011

Instrument	Calibration Procedure	Calibration Range	Frequency	Acceptance Criteria	Corrective Action	Title/Position Responsible for Corrective Action	SOP Reference
GC/MS – 8260B / 8270C/D SIM GC/MS	Minimum five-point initial calibration for all target analytes	See WS #15 for PAL/DL	Initial calibration prior to sample analysis.	System performance check compound (SPCCs) average response factor (RF) ≥ 0.050 and Percent relative standard deviation (%RSD) for RFs for CCCs $\leq 30\%$ and all other target analytes one of the following: %RSD $\leq 15\%$. or linear regression $r \geq 0.995$ or Nonlinear regression coefficient of determination $(r^2) \geq 0.99$. (min 6 points)	Correct problem then repeat initial calibration.	Lab Manager/ Analyst	DV-MS- 0010 & DV-MS- 0011
	Second- source initial calibration verification	See WS #15 for PAL/DL	Immediately following 5-point initial calibration	All analytes within <u>+</u> 20% of expected value	Correct problem then repeat initial calibration.	Lab Manager/ Analyst	
	Continuing calibration verification (CCV)	See WS #15 for PAL/DL	Daily, before sample analysis and every 12 hours of analysis time	SPCCs: average RF ≥ 0.050 % Difference/Drift for all target compounds and surrogates: ≤ 20%.	Correct problem then repeat initial calibration and reanalyze all samples since last successful CCV.	Lab Manager/ Analyst	

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Instrument	Calibration Procedure	Calibration Range	Frequency	Acceptance Criteria	Corrective Action	Title/Position Responsible for Corrective Action	SOP Reference
GC/MS – 8260B / 8270C/D SIM GC/MS	Internal Standards		Every sample/standard and blank	Retention time ±30 seconds from retention time of the midpoint standard in the Initial Calibration (ICAL) (sample/standard). Extracted Ion Current Profile area within -50% to +100% of ICAL midpoint standard.	Inspect mass spectrometer and GC for malfunctions; mandatory reanalysis of samples analyzed while system was malfunctioning (dilution of the sample may be required, see the supervisor or the technical director for advice).	Lab Manager/ Analyst	DV-MS- 0010 & DV-MS- 0011

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QAPP Worksheet #25: Analytical Instrument and Equipment Maintenance, Testing, and Inspection (UFP-QAPP Manual Section 3.2.3) (EPA 2106-G-05 Section 2.3.6)

Instrument / Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Title/Position Responsible for Corrective Action	SOP Reference
GC	Change septum, clean injection port, change or clip column, install new liner, replace column, filters and seals	Detector signals and chromatogram review	Instrument performance and sensitivity	As needed	CCV passes criteria	Re-inspect injector port, cut additional column, reanalyze CCV, recalibrate instrument	Analyst	DV-MS- 0010
GC-MS	Clean sources, maintain vacuum pumps	Tuning	Instrument performance and sensitivity	Service vacuum pumps twice per year, other maintenance as needed	Tune and CCV pass criteria	Recalibrate instrument	Analyst	DV-MS- 0011
GC-MS	Change septum, clean injection port, change or clip column, install new liner, change trap	Response factors and chromatogram review	Instrument performance and sensitivity	As needed	Tune and CCV pass criteria	Re-inspect injector port, cut additional column, reanalyze CCV, recalibrate instrument	Analyst	DV-MS- 0011

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QAPP Worksheet #26 & 27: Sample Handling, Custody, and Disposal (UFP-QAPP Manual Section 3.3) (EPA 2106-G-05 Section 2.3.3)

Sampling Organization: ECC Laboratory: Test America

Method of sample delivery (shipper/carrier): Federal Express; Hand-delivery

Number of days from reporting until sample disposal: 60

Activity	Organization and title or position of person responsible for the activity	SOP reference
Sample labeling	Samples will be assigned unique sample numbers in the field as specified in QAPP Table #18. These sample numbers will be cross-checked with the laboratory sample login receipt notification (SOP No. 3).	SOP No. 3
COC form completion		SOP No. 3
Packaging	The Site Superintendent will review the COC and inventory all samples prior to packaging and shipment or courier pickup. Glass sample containers will be placed in protective bubble wrap bags and packed with enough ice to maintain 4°C ±2°C throughout storage and shipment. COCs will be placed in a zip-seal bag and taped to the inside lid of each cooler, and the coolers sealed with strapping tape and custody seals.	SOP No. 3

Activity	Organization and title or position of person responsible for the activity	SOP reference
Shipping coordination	Samples will be shipped overnight in a sealed cooler with custody seals and the air bill number tracked, or delivered via laboratory courier (SOP No. 3 – Sample Handling and Management).	SOP No. 3
	The Site Superintendent will contact the subcontract laboratory each day following shipment of samples to verify that samples were received on time and in good condition. The laboratory will be notified in advance of the need for a Saturday sample receipt.	
	The laboratory will notify RMA-Insight of sample receipt electronically.	
Sample receipt, inspection, and log-in	Samples will be inspected for condition upon receipt; sample conditions will be recorded and forwarded to the ECC Project Chemist (see WS # 6).	SOP No. 3

Activity	Organization and title or position of person responsible for the activity	SOP reference
Sample custody and storage	COC procedures require a sample to be under custody if:	SOP No. 3
	 It is in the actual possession of the sampler It is in the view of the sampler after being in that person's physical possession The sample was in physical possession and then locked up to prevent tampering It is in a designated and identified secure area 	
	A COC record will accompany the sample at all times (SOP No. 3 – Sample Handling and Management). Field samples will be kept under strict COC in the field. The Site Superintendent will review the COC and inventory all samples prior to packaging and shipment or courier pickup. COCs will be reviewed for completeness, accuracy, and legibility. Copies of all COCs will be provided to the Project Manager on a daily basis.	
Sample disposal	Samples will be archived at the laboratory. Sample disposal will take place 90 days from the issuance of the final report.	NA

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QAPP Worksheet #28: Analytical Quality Control and Corrective Action (UFP-QAPP Manual Section 3.4 and Tables 4, 5, and 6) (EPA 2106-G-05 Section 2.3.5)

Analytical QC and corrective actions described in the following worksheets are associated with project-specific MPC. MPC are established on a project-by-project basis. The guidance documents for evaluation of MPC for this project are the EPA *Contract Laboratory Program National Functional Guidelines for Organic Superfund Data Review*, OSWER 9240.1-51, EPA 540-R-08-01, June 2008; and the EPA *Guidance on Environmental Data Verification and Data Validation (QA/G-8)*, EPA 240-R-02-004, November 2002, reissued January 2008. Additional criteria have been established based upon project-specific DQO. No validation qualification is performed absent from consideration of project DQOs.

Matrix: Soil

Analytical Group: VOCs/SVOCs

Analytical Method/SOP: SW-846 Methods 8260B/8270C/8270D SIM / DV-MS-0010, DV-MS-0011 & DV-MS-0012

QC Sample	Number/Frequency	Method/SOP Acceptance Criteria	Corrective Action	Title/Position of Person Responsible for Corrective Action	Project-Specific MPC
Method	1/Preparatory Batch	No Target	If sufficient sample is	Analyst /	All analytes in the method blank
Blank	(20 samples)	Compounds> 1/2	available, reanalyze	Section	must be less than ½ the
		reporting limit	samples. Qualify	Supervisor	quantitation limit or 1/5 of the
		(RL); and $> 1/10$	data as needed.	_	PALs on WS#15, whichever is
		the amount in any	Report results if		greater; corollary detections in
		sample or 1/10 the	sample results >10x		field samples may be qualified as
		regulatory limit	blank result or		non-detect (U) for concentrations \leq
		(whichever is	sample results non-		5x the concentration in the blank
		greater). No	detect.		(10x for common lab
		common lab			contaminants).
		contaminants >RL.			

QC Sample	Number/Frequency	Method/SOP Acceptance Criteria	Corrective Action	Title/Position of Person Responsible for Corrective Action	Project-Specific MPC
Laboratory Control Sample	1/Preparatory Batch (20 samples)	Refer to the 8260B Attachment for laboratory control sample (LCS) control limits.	If sufficient sample is available, reanalyze samples. Qualify data as needed.	Analyst / Section Supervisor	Laboratory limits; evaluate direction of bias, effect on detected concentrations vs. PAL, and apply professional judgment for impact on data for project objectives. Also See WS #12.
Matrix Spike / Matrix Spike Duplicate	1/OWS Site	Refer to the 8260B Attachment for LCS control limits.	Determine root cause; flag matrix spike/matrix spike duplicate (MS/MSD) data; discuss in narrative.	Analyst / Section Supervisor	Laboratory limits; evaluate direction of bias, effect on detected concentrations vs. PAL, and apply professional judgment for impact on data for project objectives. Also see WS #12.
Surrogates	Every sample	Refer to the 8260B Attachment for Surrogate control limits.	Check calculations and instrument performance; recalculate, reanalyze.	Analyst / Section Supervisor	Laboratory limits; evaluate direction of bias, effect on detected concentrations vs. PAL, and apply professional judgment for impact on data for project objectives.

QAPP Worksheet #29: Project Documents and Records

(UFP-QAPP Manual Section 3.5.1) (EPA 2106-G-05 Section 2.2.8)

	Sample Collection and Field Records						
Record	Generation	Verification	Storage location/archival				
Field logbook or data collection sheets	Field Team Leader (John	Project Chemist (Dianne	Electronic Project File				
	Ryder)	McNeill	(electronic/portable document				
			format [PDF])				
COC Forms	Field Team Leader (John	Project Chemist (Dianne	Electronic Project File				
	Ryder)	McNeill	(electronic/PDF)				
Air bills	Field Team Leader (John	ECC PM (Jon Vail)	Electronic Project File				
	Ryder)		(electronic/PDF)				
Contractor Daily QC Reports	Field Team Leader (John	ECC PM (Jon Vail)	Electronic Project File				
	Ryder)		(electronic/PDF)				
Deviations	Field Team Leader (John	ECC PM (Jon Vail)	Electronic Project File				
	Ryder)		(electronic/PDF)				
Corrective Action Reports	Field Team Leader (John	ECC PM (Jon Vail)	Electronic Project File				
	Ryder)		(electronic/PDF)				
Correspondence	Project Task Manager/ECC	RMA-Insight Senior PM	Electronic Project File				
	PM (Jon Vail)	(Mittra Fatthipour)	(electronic/PDF)				

Project Assessments						
Record	Generation	Verification	Storage location/archival			
Field audit checklists	Project Task Manager/ECC	Field Team Leader (John	Electronic Project File			
	PM (Jon Vail)	Ryder)	(electronic/PDF)			
Data verification checklists	Project Chemist (Dianne	Field Team Leader (John	Electronic Project File			
	McNeill	Ryder)	(electronic/PDF)			
Data validation report	Project Chemist (Dianne	Field Team Leader (John	Electronic Project File			
_	McNeill	Ryder)	(electronic/PDF)			
Data usability assessment report	Project Chemist (Dianne	Field Team Leader (John	Electronic Project File			
_	McNeill	Ryder)	(electronic/PDF)			

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Laboratory Records					
Record	Generation	Verification	Storage location/archival ¹		
Laboratory QC Summary and Data Package	TA	Project Chemist (Dianne McNeill	ECC DB/EDMS; electronic project file (electronic/PDF)		
Laboratory EDD	TA	Project Chemist (Dianne McNeill	ECC DB/EDMS		

¹ Laboratory EDDs will be uploaded directly to the ECC Database/Electronic Data Management System and checked for compliance; uploading compliant EDDs is the responsibility of the laboratory. Ensuring laboratory data packages are filed in ECC's project file is the responsibility of the ECC Project Chemist.

Laboratory Data Deliverables					
Record	VOCs	SVOCs			
Narrative	X	X			
COC	X	X			
Summary Results	X	X			
QC Results	X	X			
Chromatograms	X	X			

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QAPP Worksheet #31, 32 & 33: Assessments and Corrective Action (UFP-QAPP Manual Sections 4.1.1 and 4.1.2) (EPA 2106-G-05 Section 2.4 and 2.5.5)

Assessments:

Assessment Type	Responsible Party & Organization	Number/Frequency	Estimated Dates	Assessment Deliverable	Deliverable Due Date
Field Sampling technical systems audit (TSA)	Field Team Lead ECC	One each on first day of soil sampling, bench-test initiation, and groundwater sampling episodes		Memorandum of acceptability of procedures or need for filed change delivered via e-mail to ECC PM/Task Manager and RMA- Insight QCM	24 hours following assessment for e-mail to ECC PM; additional 24 hours for notification of AFCEC PM
Laboratory Audit/Review of SOPs and representative data packages	Project Chemist ECC	Prior to start of on-site analytical work		Memorandum of acceptability of procedures or need for filed change delivered via e-mail to ECC PM/Task Manager and RMA-Insight QCM; ECC PM will report results to the AFCEC PM	48 hours following assessment for e-mail to ECC PM; additional 24 hours for notification of AFCEC PM
Project-specific	AFCEC in	As determined by	As determined by	PT Deficiency Report	48 hours following
proficiency testing (PT) samples	coordination with ECC Project Chemist	AFCEC	AFCEC		receipt of PT results

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Assessment Response and Corrective Action:

Assessment Type	Responsibility for Responding to Assessment Findings	Assessment Response Documentation	Time Frame for Response	Responsibility for Implementing Corrective Action	Responsible for Monitoring Corrective Action implementation
Field Sampling TSA	Field Task Leader	Field Sampling	24 hours from receipt	Field Team Leader	RMA-Insight Quality
		Corrective Action Response	of Memorandum		Manager
Laboratory	Laboratory Quality	On-site Analytical	48 hours from receipt	Laboratory Technical	ECC Project Chemist
Audit/Review of	Assurance Manager	Corrective Action	of Memorandum and	Director/QAM	
SOPs and	(QAM)	Response	before further		
representative data			analyses can be		
packages			conducted		
Project-specific PT	Off-Site Laboratory	PT Deficiency	7 days following	Laboratory Technical	ECC Project Chemist
samples	QAM	Memorandum	receipt of PT	Director/QAM	
			Deficiency Report		
			and before analysis		
			field samples		

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QAPP Worksheet #34: Data Verification and Validation Inputs (UFP-QAPP Manual Section 5.2.1 and Table 9) (EPA 2106-G-05 Section 2.5.1)

Item	Description	Verification (completeness)	Validation (conformance to specifications)				
	Planning Documents/Records						
1	Approved QAPP	X					
2	Contract	X					
4	Field SOPs	X					
5	Laboratory SOPs	X					
	Field Records						
6	Field logbooks	X	X				
7	Equipment calibration records	X	X				
8	Chain-of-Custody Forms	X	X				
9	Sampling diagrams/surveys	X	X				
10	Drilling logs	X	X				
11	Geophysics reports	X	X				
12	Relevant Correspondence	X	X				
13	Change orders/deviations	X	X				
14	Field audit reports	X	X				
15	Field corrective action reports	X	X				
16	Sampling field forms	X	X				
	Analytical Data Pack	age					
17	Cover sheet (laboratory identifying information)	X	X				
18	Case narrative	X	X				
19	Internal laboratory chain-of-custody	X	X				
20	Sample receipt records	X	X				
21	Sample chronology (e.g. dates and times of receipt, preparation, & analysis)	X	X				
22	Communication records	X	X				
23	Project-specific PT sample results	X	X				
24	LOD/LOQ establishment and verification	X	X				
25	Standards Traceability	X	X				
26	Instrument calibration records	X	X				
27	Definition of laboratory qualifiers	X	X				
28	Results reporting forms	X	X				
29	QC sample results	X	X				
30	Corrective action reports	X	X				
31	Raw data	X	X				
32	Electronic data deliverable	X	X				

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QAPP Worksheet #35: Data Verification Procedures (UFP-QAPP Manual Section 5.2.2) (EPA 2106-G-05 Section 2.5.1)

Records Reviewed	Requirement Documents	Process Description	Responsible Person, Organization
Field logbook	QAPP, SOP Field 02	Verify that records are present and complete for each day of field activities. Verify that all planned samples including field QC samples were collected and that sample collection locations are documented. Verify that meteorological data were provided for each day of field activities. Verify that changes/exceptions are documented and were reported in accordance with requirements. Verify that any required field monitoring was performed and results are documented.	Daily — Project Manager At conclusion of field activities — Project QA Manager
Chain-of-Custody forms	QAPP, SOP Field 02	Verify the completeness of chain-of-custody records. Examine entries for consistency with the field logbook. Check that appropriate methods and sample preservation have been recorded. Verify that the required volume of sample has been collected and that sufficient sample volume is available for QC samples (e.g., MS/MSD). Verify that all required signatures and dates are present. Check for transcription errors.	Daily — Field Crew Chief At conclusion of field activities — Project Chemist
Laboratory Deliverable	QAPP	Verify that the laboratory deliverable contains all records specified in the QAPP. Check sample receipt records to ensure sample condition upon receipt was noted, and any missing/broken sample containers were noted and reported according to plan. Compare the data package with the COCs to verify that results were provided for all collected samples. Review the narrative to ensure all QC exceptions are described. Check for evidence that any required notifications were provided to project personnel as specified in the QAPP. Verify that necessary signatures and dates are present.	Before release — Laboratory QAM Upon receipt - Project Chemist
Audit Reports, Corrective Action Reports	QAPP	Verify that all planned audits were conducted. Examine audit reports. For any deficiencies noted, verify that corrective action was implemented according to plan.	Project QAM

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QAPP Worksheet #36: Data Validation Procedures (UFP-QAPP Manual Section 5.2.2) (EPA 2106-G-05 Section 2.5.1)

Data Validator: ECC Chemists, under direction of ECC Project Chemist
Data will not be validated by project field personnel or PM, or by any personnel reporting directly to such personnel

Analytical Group/Method:	All site characterization and long term
	monitoring data
Data deliverable requirements:	ECC DB/DMS
Analytical specifications:	See WK 23
Measurement performance criteria:	See WS #10, #12, #15, and #20
Percent of data packages to be validated (Level	100% of laboratory data for site
II – QC summary review including surrogates,	characterization and/or confirmation of no
sample receipt and field documentation	action required
review):	
Percent of data packages to be validated (Level	100% of laboratory data
III – Level II + internal standard and	
calibration review):	
Percent of raw data reviewed (Level IV =	10% of laboratory data
Level III plus recalculation of selected	
detections [see below], calibration results,	
recoveries and chromatography review):	
Percent of results to be recalculated:	10% of laboratory data
Validation procedure:	EPA National Function Guidelines
Validation Qualifiers	U, UJ, R, or J

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QAPP Worksheet #37: Data Usability Assessment (UFP-QAPP Manual Section 5.2.3 including Table 12) (EPA 2106-G-05 Section 2.5.2, 2.5.3, and 2.5.4)

Identify personnel (organization and position/title) responsible for participating in the data usability assessment:

Project Manager Project QCM Geologist/Hydrogeologist Project Chemist Field Task Leader

Describe how the usability assessment will be documented:

Data usability recommendations will be provided in data validation reports for recommendations to the project team. Final reports will provide a summary of data usability incorporating data validation findings and general assessment of data representativeness and accuracy based upon temporal and spatial correlation of data observations and trends being consistent with the site CSM.

Step 1	Review the project's objectives and sampling design			
	The project team will review the project DQOs and measurement performance criteria to ensure they still support the project objectives and determine if the CSM needs to be revised based upon the most current site data.			
Step 2	Review the data verification and data validation outputs			
	The applicable members of the project team (WS #35) will prepare and/or review project QA/QC reports. Summarized data will be evaluated for patterns, trends, and anomalies not consist with past observations or the CSM.			
	Review deviations from planned activities (e.g., number and locations of samples, holding time exceedances, damaged samples, and SOP deviations) and determine their effects on the data usability.			

Step 3	Verify the assumptions of the selected statistical method
	Samples may be collected to support EPC calculations. EPA Region 8 EPC calculations will be reviewed to verify assumptions for the statistical method.
Step 4	Implement the statistical method
	In the event that a point-by-point comparison does not show that all of the constituents detected are below their respective residential/unrestricted use levels and soil concentrations protective of groundwater (collectively referred to as NFA cleanup levels), then the derivation of an exposure point concentration may be used. In risk assessment terms, the level of contamination that a person may be exposed to over a given area is called the "exposure point concentration" or EPC. This concentration is a conservative estimate of the average chemical concentration in an environmental medium and is calculated for each individual exposure area within a site.
Step 5	Document data usability and draw conclusions
	Data usability recommendations and limitations will be provided in data validation reports for recommendations to the Peterson AFM and ECC PMs Data collected that are not qualified as rejected will be used to fully characterize the extent of soil contamination at Sites OW011 and OW012. Comparing data to EPA RSL-Rs and/or utilizing data for EPC calculations will facilitate revision of the CSM for both OWS sites.





STATE OF COLORADO

Department of Public Health and Environment

Under Primacy Agreement with the United States Environmental Protection Agency Pursuant to the Safe Drinking Water Regulations, 40CFR, Part 141

Certifies

TestAmerica - Denver 4955 Yarrow Street Arvada, CO 80002

is in compliance with the criteria and procedures of the EPA Manual for the Certification of Laboratories Analyzing Drinking Water. The laboratory may perform Chemical analyses on public drinking water for the following parameters:

Trace Metals (Limited EPA-200.7), Mercury, Nitrate, Nitrite, Fluoride, EDB, DBCP.

Approved analytes and methods are delineated on certification list of October 1, 2011.

EFFECTIVE:

October 1, 2012 through September 30, 2013.



David A. Butcher, Director Laboratory Services Division



STATE OF COLORADO CHEMISTRY CERTIFICATION STATUS SAFE DRINKING WATER ACT

Name: TestAmerica - Denver

4955 Yarrow Street Arvada, CO 80002

Date: October 1, 2012



STATUS	TRACE METALS	METHODS	STATUS	CARBAMATES	<u>METHODS</u>
(A)	Antimony		(N)	Carbofuran	
(A)	Arsenic	EDA 000 7	(N)	Oxamyl (Vydate)	
. (A)	Barium	EPA-200.7 EPA-200.7			
(A) (A)	Beryllium Cadmium	EPA-200.7 EPA-200.7		HERBICIDES	
(A) (A)	Chromium	EPA-200.7		HERDICIBES	
(A)	Copper	EPA-200.7	(N)	2,4-D	
(A)	Lead		(N)	2,4,5-TP	
(A)	Mercury	EPA-245.1	(N)	Dalapon	
(A)	Nickel	EPA-200.7	(N)	Dinoseb	
(A)	Selenium		(N)	Pentachlorophenol	
(A)	Thallium		(N)	Picloram	
	URANIUM			PCBs, as	
(A)	Uranium (by ICP-MS)	EPA-200.8	(N)	Decachlorobiphenyl	
(A)	Orallium (by ICF-INIS)	LF A-200.0	(14)	Decacilloropiphenyi	
	NITRATE / NITRITE / FLUOF	RIDE		PAH	
			(N)	Benzo(a)pyrene	
(A)	Nitrate-N	EPA-300.0			
(A)	Nitrite-N	EPA-300.0			-
(A)	Fluoride	EPA-300.0			
	PESTICIDES	,		ADIPATES / PHTHALATES	
(N)	Alachlor		(N)	Bis-(2-ethylhexyl) Adipate	
(N)	Atrazine		(N)	Bis-(2-ethylhexyl) Phthalate	
(N)	Chlordane	·································			
(N)	Endrin			•	
(N)	Heptachlor				
(N)	Heptachlor epoxide				
(N)	Hexachlorobenzene			(A) = Approved / Certified	
(N)	Hexachlorocyclopentadiene			(N) = Not Certified	
(N)	Lindono			(P) = Provisionally Certified	
	Lindane			•	
(N)	Methoxychlor	***************************************		(I) = Interim	
(N) (N) (N)				•	

STATE OF COLORADO CHEMISTRY CERTIFICATION STATUS SAFE DRINKING WATER ACT

Name: TestAmerica - Denver

4955 Yarrow Street Arvada, CO 80002

<u>Date</u>: October 1, 2012



STATUS	TRIHALOMETHANES	METHODS	STATUS	MISCELLANEOUS	METHODS
<u>(N)</u>	Total Trihalomethanes		(N)	Diquat	
(N)	Bromodichloromethane		(N)	Endothall	
(N)	Bromoform		(N)	Glyphosate	
(N)	Chlorodibromomethane		(N)	Dioxin	
(N)	Chloroform		(N)	Total Organic Carbon	
			(N)	Asbestos	
			(N)	Total Cyanide	
	VOLATILE ORGANICS		(A)	Bromide	EPA-300.0
4			(N)	Bromate	
	V1 - DBCP/EDB		(N)	Chlorite	
(A) (A)	1,2-Dibromochloropropane Ethylene dibromide	EPA-504.1 EPA-504.1			
(N)	<u>V2</u> - Vinyl Chloride			HALOACETIC ACIDS	
<u>(N)</u>	<u>V3</u> - Regulated VOCs		<u>(N)</u>	<u>HAA-5</u>	
(N)	Benzene	***********	(N)	Chloroacetic Acid	MANAGE = = = = = = = = = = = = = = = = = = =
(N)	Carbon tetrachloride		(N)	Dichloroacetic Acid	
(N)	1,2-Dichlorobenzene		(N)	Trichloroacetic Acid	
(N)	1,2-Dichloroethane		(N)	Bromoacetic Acid	
(N)	1,1-Dichloroethylene		(N)	Dibromoacetic Acid	
(N)	Trichloroethylene				
(N)	Chlorobenzene				
(N)	1,4-Dichlorobenzene			•	
(N)	c-1,2-Dichloroethylene				
(N)	t-1,2-Dichloroethylene				
(N)	1,2-Dichloropropane				
(N)	Ethylbenzene			(A) = Approved / Certified	
(N)	Styrene			(N) = Not Certified	
(N)	Tetrachloroethylene			(P) = Provisionally Certified	
(N)	Toluene			(I) = Interim	
(N)	1,1,1-Trichloroethane				
(N)	Total Xylenes				
(N)	Dichloromethane				
(N)	1,2,4-Trichlorobenzene				
(N)	1,1,2-Trichloroethane				

STATE OF COLORADO

John W. Hickenlooper, Governor Christopher E. Urbina, MD, MPH Executive Director and Chief Medical Officer

Dedicated to protecting and improving the health and environment of the people of Colorado

Laboratory Services Division 8100 Lowry Blvd. Denver, Colorado 80230-6928 (303) 692-3090

http://www.cdphe.state.co.us/lr



October 4, 2012

Mr. John Morris **Test America Laboratories, Inc.** 4955 Yarrow Street Arvada. CO 80002

RE: Chemistry Certification

Dear Mr. Morris:

Enclosed is your new Colorado Department of Public Health and Environment Safe Drinking Water (SDW) Chemistry Certificate along with your Chemistry status report dated October 1, 2012, which is effective through September 30, 2013, unless suspended or revoked prior to that date, and is based upon findings from the August 16, 2012 onsite assessment, your acceptable Plan of Correction dated September 24, 2012, and successful participation in recent Water Supply Proficiency Testing Studies for your approved parameters.

This certification must be renewed by September 2013. Routine on-site assessments are conducted every two years (biennial) unless the certifications are modified. However, it is the laboratory's responsibility to submit a renewal application and annual certification fee. Our renewal application and requirements are now available at: www.coloradowaterlabs.us.

If you have any questions, or if there are changes that may affect your certification status, you can reach me at (303) 692-3045.

Best Regards,

Ben Chouaf, Certification Officer Laboratory Services Division ben.chouaf@state.co.us

Attachment, as stated



SCOPE OF ACCREDITATION TO ISO/IEC 17025:2005

TESTAMERICA DENVER 4955 Yarrow Street Arvada, CO 80002 Karen Kuoppala Phone: 303-736-1203 www.testamericainc.com

ENVIRONMENTAL

Valid To: October 31, 2013 Certificate Number: 2907.01

In recognition of the successful completion of the A2LA evaluation process, (including an assessment of the laboratory's compliance with ISO IEC 17025:2005, the 2003 NELAC Chapter 5 Standard, and the requirements of the DoD Environmental Laboratory Accreditation Program (DoD ELAP) as detailed in the current DoD Quality Systems Manual for Environmental Laboratories) accreditation is granted to this laboratory to perform recognized EPA methods using the following testing technologies and in the analyte categories identified below:

Testing Technologies

Atomic Absorption/ICP-AES Spectrometry, ICP/MS, Gas Chromatography, Gas Chromatography/Mass Spectrometry, Gravimetry, High Performance Liquid Chromatography, Ion Chromatography, Misc.- Electronic Probes (pH, O₂), Oxygen Demand, Hazardous Waste Characteristics Tests, Spectrophotometry (Visible), Spectrophotometry (Automated), Titrimetry, Total Organic Carbon, Total Organic Halide

Parameter/Analyte	Non-Potable Water	Solid Hazardous Waste	Solid Hazardous Waste		
		(Water)	(Solid)		
<u>Metals</u>					
Aluminum		EPA 6010B/6010C	EPA 6010B/6010C		
Antimony		EPA	EPA		
		6010B/6010C/6020/6020A	6010B/6010C/6020/6020A		
Arsenic		EPA	EPA		
		6010B/6010C/6020/6020A	6010B/6010C/6020/6020A		
Barium		EPA	EPA		
		6010B/6010C/6020/6020A	6010B/6010C/6020/6020A		
Beryllium		EPA	EPA		
		6010B/6010C/6020/6020A	6010B/6010C/6020/6020A		
Boron		EPA 6010B/6010C	EPA 6010B/6010C		
Cadmium		EPA	EPA		
		6010B/6010C/6020/6020A	6010B/6010C/6020/6020A		
Calcium		EPA 6010B/6010C	EPA 6010B/6010C		
Chromium		EPA	EPA		
		6010B/6010C/6020/6020A	6010B/6010C/6020/6020A		
Cobalt		EPA	EPA		
		6010B/6010C/6020/6020A	6010B/6010C/6020/6020A		
Copper		EPA	EPA		
	\mathcal{O}_{L}	60)/9 B/6010C/6020/6020A	6010B/6010C/6020/6020A		
Leter Mhyer					
(A2LA Cert. No. 2907.01) Revised 10/12/2011 Page 1 of 13					

Parameter/Analyte	Non-Potable Water	Solid Hazardous Waste	Solid Hazardous Waste
		(Water)	(Solid)
Iron		EPA 6010B/6010C	EPA 6010B/6010C
Lead		EPA	EPA
		6010B/6010C/6020/6020A	6010B/6010C/6020/6020A
Lithium		EPA 6010B/6010C	EPA 6010B/6010C
Magnesium		EPA 6010B/6010C	EPA 6010B/6010C
Manganese		EPA	EPA
		6010B/6010C/6020/6020A	6010B/6010C/6020/6020A
Mercury		EPA 7470A	EPA /7471A/7471B
Molybdenum		EPA	EPA
, J		6010B/6010C/6020/6020A	6010B/6010C/6020/6020A
Nickel		EPA	EPA
		6010B/6010C/6020/6020A	6010B/6010C/6020/6020A
Potassium		EPA 6010B/6010C	EPA 6010B/6010C
Selenium		EPA	EPA
Selement		6010B/6010C/6020/6020A	6010B/6010C/6020/6020A
Silica		EPA 6010B/6010C	EPA 6010B/6010C
Silicon		EPA 6010B/6010C	EPA 6010B/6010C
Silver		EPA	EPA
Silver		6010B/6010C/6020/6020A	6010B/6010C/6020/6020A
Sodium		EPA 6010B/6010C	EPA 6010B/6010C
Strontium		EPA 6010B/6010C	EPA 6010B/6010C
Thallium		EPA 0010B/0010C	EPA 0010B/0010C
Hamum		6010B/6010C/6020/6020A	6010B/6010C/6020/6020A
Tin		EPA 6010B/6010C	
			EPA 6010B/6010C
Titanium		EPA 6010B/6010C	EPA 6010B/6010C
Vanadium		EPA	EPA
7.		6010B/6010C/6020/6020A	6010B/6010C/6020/6020A
Zinc		EPA	EPA
		6010B/6010C/6020/6020A	6010B/6010C/6020/6020A
NT			
Nutrients Ni	D 1 1 .:	D 1 1 / /EDA	D 1 1 / FD4
Nitrate (as N)	By calculation	By calculation/ EPA	By calculation/ EPA
		9056/9056A	9056/9056A
Nitrate-nitrite (as N)	EPA 353.2	EPA 353.2/ EPA	EPA 9056/9056A
		9056/9056A	
Nitrite (as N)	SM 4500-NO2 B	SM 4500-NO2 B/ EPA	EPA 9056/9056A
		9056/9056A	
Orthophosphate (as P)		EPA 9056/9056A	EPA 9056/9056A
Total phosphorus		EPA 6010B/6010C	EPA 6010B/6010C
Demands			
Total organic carbon		EPA 9060 /9060A	EPA 9060 /9060A
Total organic halides		EPA 9020B	
Wet Chemistry			
Alkalinity	SM 2320 B	SM 2320 B	SM 2320 B
Ammonia	EPA 350.1	EPA 350.1	
Biological Oxygen Demand	SM 5210B	SM 5210B	
Bromide		EPA 9056/9056A	EPA 9056/9056A
Diomiac			
Total organic carbon		EPA 9060/9060A	EPA 9060/9060A
		EPA 9060/9060A EPA 9056/9056A	EPA 9060/9060A EPA 9056/9056A

Peter Mhye Page 2 of 13

Parameter/Analyte	Non-Potable Water	Solid Hazardous Waste	Solid Hazardous Waste
		(Water)	(Solid)
Conductivity		EPA 9050/EPA 9050A	EPA 9050/EPA 9050A
Cyanide		EPA 9010B/9012A/9012B	EPA 9010B/9012A/9012B
Ferrous Iron	SM 3500 Fe B, D	SM 3500 Fe B, D	
Fluoride		EPA 9056/9056A	EPA 9056/9056A
Hexavalent Chromium	EPA 7196A	EPA 7196A	EPA 7196A/3060A
pH		EPA 9040B/9045C	EPA 9040B/9045C
Oil and Grease (HEM and	EPA 1664A	EPA 1664A	9071B
SGT-HEM)		2111100111	50,12
Percent moisture			ASTM D2216
Perchlorate		EPA 6860	EPA 6860
Phenols		EPA 9066	EPA 9066
Solids, Total	SM 2540 B	SM 2540 B	SM 2540 B
Solids, Total Suspended	SM 2540 D	SM 2540 D	SM 2540 D
Solids, Total Dissolved	SM 2540 C	SM 2540 C	SM 2540 C
Sulfate	5W1 2540 C	EPA 9038/9056/9056A	EPA 9038/9056/9056A
Sulfide, Total		EPA 9038/9036/9036A EPA 9034	EPA 9038/9036/9036A EPA 9034
Sulfide, Total Sulfide		EPA 9034 EPA 9030B	EPA 9034 EPA 9030B
			EPA 9030B
Total Kjeldahl Nitrogen	EPA 351.2	EPA 351.2	
Purgeable Organics (volatiles)			
Acetone		EPA 8260B	EPA 8260B
Acetonitrile		EPA 8260B	EPA 8260B
Acrolein		EPA 8260B	EPA 8260B
Acrylonitrile		EPA 8260B	EPA 8260B
Allyl Chloride		EPA 8260B	EPA 8260B
Benzene		EPA 8260B/8021B/AK101	EPA 8260B/8021B/AK101
Bromobenzene		EPA 8260B	EPA 8260B
Bromochloromethane		EPA 8260B	EPA 8260B
Bromodichloromethane		EPA 8260B	EPA 8260B
Bromoform		EPA 8260B	EPA 8260B
Bromomethane		EPA 8260B	EPA 8260B
2-Butanone		EPA 8260B	EPA 8260B
n-Butyl alcohol	<u> </u>	EPA 8260B/8015B/8015C	EPA 8260B/8015B/8015C
n-Butyl aconor n-Butylbenzene		EPA 8260B	EPA 8260B
Sec-Butylbenzene		EPA 8260B	EPA 8260B
Tert-Butylbenzene		EPA 8260B	EPA 8260B
Carbon disulfide		EPA 8260B	EPA 8260B
Carbon tetrachloride		EPA 8260B	EPA 8260B
Chlorobenzene		EPA 8260B / 8021B	EPA 8260B / 8021B
			1
2-Chloro-1,3-butadiene		EPA 8260B	EPA 8260B
Chloroethane		EPA 8260B	EPA 8260B
2-Chloroethyl vinyl ether		EPA 8260B	EPA 8260B
Chloroform		EPA 8260B	EPA 8260B
1-Chlorohexane		EPA 8260B	EPA 8260B
Chloromethane		EPA 8260B	EPA 8260B
Chloroprene		EPA 8260B	EPA 8260B
3-Chloroprene		EPA 8260B	EPA 8260B
4-Chlorotoluene		EPA 8260B	EPA 8260B
2-Chlorotoluene		EPA 8260B	EPA 8260B
Cyclohexane		EPA 8260B	EPA 8260B
Cyclohexanone		EPA 8260B	EPA 8260B

Tetu Mhyu Page 3 of 13

Parameter/Analyte	Non-Potable Water	Solid Hazardous Waste	Solid Hazardous Waste
		(Water)	(Solid)
Dibromochloromethane		EPA 8260B	EPA 8260B
1,2-Dibromo-3-	EPA 504	EPA 504/ EPA 8260B/8011	EPA 8260B/8011
chloropropane (DBCP)			
Dibromochloromethane		EPA 8260B	EPA 8260B
Dichlorodifluoromethane		EPA 8260B	EPA 8260B
Dibromomethane		EPA 8260B	EPA 8260B
1,2 Dibromoethane (EDB)	EPA 504	EPA 504/ EPA 8260B/8011	EPA 8260B/8011
1,2-Dichlorobenzene		EPA 8260B/8021B	EPA 8260B/8021B
1,3-Dichlorobenzene		EPA 8260B/8021B	EPA 8260B/8021B
1,4-Dichlorobenzene		EPA 8260B/8021B	EPA 8260B/8021B
cis-1,4-Dichloro-2-butene		EPA 8260B	EPA 8260B
trans-1,4-Dichloro-2-butene		EPA 8260B	EPA 8260B
1,1-Dichloroethane		EPA 8260B	EPA 8260B
1,2-Dichloroethane		EPA 8260B	EPA 8260B
1.1-Dichloroethene		EPA 8260B	EPA 8260B
,			
1,2-Dichloroethene		EPA 8260B	EPA 8260B
cis-1,2-Dichloroethene		EPA 8260B	EPA 8260B
trans-1,2-Dichloroethene		EPA 8260B	EPA 8260B
Dichlorofluoromethane		EPA 8260B	EPA 8260B
1,2-Dichloropropane		EPA 8260B	EPA 8260B
1,3-Dichloropropane		EPA 8260B	EPA 8260B
2,2-Dichloropropane		EPA 8260B	EPA 8260B
1,1-Dichloropropene		EPA 8260B	EPA 8260B
1,3-Dichloropropene		EPA 8260B	EPA 8260B
cis-1,3-Dichloropropene		EPA 8260B	EPA 8260B
trans-1,3-Dichloropropene		EPA 8260B	EPA 8260B
Diethyl ether		EPA 8260B	EPA 8260B
Di-isopropylether		EPA 8260B	EPA 8260B
1,4-Dioxane		EPA 8260B/8260B SIM	EPA 8260B/8260B SIM
Ethanol		EPA 8260B/8015B/8015C	EPA 8260B/8015B/8015C
Ethyl acetate		EPA 8260B	EPA 8260B
Ethyl benzene		EPA 8260B/8021B/AK101	EPA 8260B/8021B/AK101
Ethyl methacrylate		EPA 8260B	EPA 8260B
Ethylene Glycol		EPA 8015C	EPA 8015C
Gas Range Organics (GRO)		EPA	EPA
		8015B/8015C/AK101/8015D	8015B/8015C/AK101/8015D
Hexane		EPA 8260B	EPA 8260B
2-Hexanone		EPA 8260B	EPA 8260B
Hexachlorobutadiene		EPA 8260B	EPA 8260B
Isobutyl alcohol (2-Methyl-		EPA 8260B/8015B/8015C	EPA 8260B/8015B/8015C
1-propanol)			
Isopropyl alcohol		EPA 8260B	EPA 8260B
Isopropylbenzene		EPA 8260B	EPA 8260B
1,4-Isopropyltoluene		EPA 8260B	EPA 8260B
Iodomethane		EPA 8260B	EPA 8260B
Methacrylonitrile		EPA 8260B	EPA 8260B
Methanol		EPA 8015B/8015C	EPA 8015B/8015C
Methyl acetate		EPA 8013B/8013C EPA 8260B	EPA 8260B
Methyl cyclohexane		EPA 8260B	EPA 8260B
• •		EPA 8260B EPA 8260B	EPA 8260B
Methylene chloride			
Methyl ethyle ketone		EPA 8260B	EPA 8260B
(MEK)	i d 10/12/2011	1 De M	<u> </u>

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s Waste
<u> </u>
21B
-12
021B/AK101
<u>/21B</u> //111101
11
21B/AK101
21B/AK101
21B/AK101
70D/8270SIM

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Parameter/Analyte	Non-Potable Water	Solid Hazardous Waste	Solid Hazardous Waste
<u>Farameter/Anaryte</u>	Non-Potable Water	(Water)	(Solid)
Aganamhthrilana	_	EPA	EPA 8270C/8270D/8270SIM
Acenaphthylene		8270C/8270D/8270SIM	EPA 82/0C/82/0D/82/0SIM
Acceptant		EPA 8270C/8270D	EDA 9270C/9270D
Acetophenone			EPA 8270C/8270D
2-Acetylaminofluorene		EPA 8270C/8270D	EPA 8270C/8270D
Alachlor		EPA 8270C/8270D	EPA 8270C/8270D
4-Aminobiphenyl		EPA 8270C/8270D	EPA 8270C/8270D
Aniline		EPA 8270C/8270D	EPA 8270C/8270D
Anthracene		EPA	EPA 8270C/8270D/8270SIM
		8270C/8270D/8270SIM	
Aramite		EPA 8270C/8270D	EPA 8270C/8270D
Atrazine		EPA 8270C/8270D	EPA 8270C/8270D
Azobenzene		EPA 8270C/8270D	EPA 8270C/8270D
Benzaldehyde		EPA 8270C/8270D	EPA 8270C/8270D
Benzidine		EPA 8270C/8270D	EPA 8270C/8270D
Benzoic acid		EPA 8270C/8270D	EPA 8270C/8270D
Benzo (a) anthracene		EPA	EPA 8270C/8270D/8270SIM
		8270C/8270D/8270SIM	
Benzo (b) fluoranthene		EPA	EPA 8270C/8270D/8270SIM
		8270C/8270D/8270SIM	
Benzo (k) fluoranthene		EPA	EPA 8270C/8270D/8270SIM
Denze (k) Hustaninene		8270C/8270D/8270SIM	2111 027 0 07 027 027 03111
Benzo (ghi) perylene		EPA	EPA 8270C/8270D/8270SIM
Benzo (gm) peryiene		8270C/8270D/8270SIM	E171 0270 C/0270 D/0270 S1111
Benzo (a) pyrene	<u> </u>	EPA	EPA 8270C/8270D/8270SIM
Benzo (a) pyrene		8270C/8270D/8270SIM	El IX 02/0C/02/0D/02/05livi
Benzyl alcohol	<u> </u>	EPA 8270C/8270D	EPA 8270C/8270D
Bis (2-chloroethoxy)		EPA 8270C/8270D	EPA 8270C/8270D
methane		EFA 82/0C/82/0D	EFA 82/0C/82/0D
Bis (2-chloroethyl) ether	1	EPA 8270C/8270D	EPA 8270C/8270D
Bis (2-chloroisopropyl)		EPA 8270C/8270D EPA 8270C/8270D	EPA 8270C/8270D
		EPA 82/0C/82/0D	EPA 82/0C/82/0D
ether (2,2'Oxybis(1-			
chloropropane)		EPA 8270C/8270D	EPA 8270C/8270D
Bis (2-ethylhexyl) phthalate			
4-Bromophenyl phenyl		EPA 8270C/8270D	EPA 8270C/8270D
ether		ED 4 00700/0070D	EDA 0270C/0270D
Butyl benzyl phthalate		EPA 8270C/8270D	EPA 8270C/8270D
2-sec-Butyl-4,6-		EPA 8270C/8270D	EPA 8270C/8270D
dinitrophenol		FD 4 0250 G (0250 D	ED A COROCIOSEO
Carbazole		EPA 8270C/8270D	EPA 8270C/8270D
4-Chloroanilene		EPA 8270C/8270D	EPA 8270C/8270D
Chlorobenzilate		EPA 8270C/8270D	EPA 8270C/8270D
4-Chloro-3-methylphenol		EPA 8270C/8270D	EPA 8270C/8270D
1-Chloronaphthalene		EPA 8270C/8270D	EPA 8270C/8270D
2-Chloronaphthalene		EPA 8270C/8270D	EPA 8270C/8270D
2-Chlorophenol		EPA 8270C/8270D	EPA 8270C/8270D
4-Chlorophenyl phenyl		EPA 8270C/8270D	EPA 8270C/8270D
ether			
Chrysene		EPA	EPA 8270C/8270D/8270SIM
		8270C/8270D/8270SIM	
Cresols		EPA 8270C/8270D	EPA 8270C/8270D
Diallate		EPA 8270C/8270D	EPA 8270C/8270D
Dialiate		EPA 82/UC/82/UD	EPA 82/0C/82/0D

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Parameter/Analyte	Non-Potable Water	Solid Hazardous Waste	Solid Hazardous Waste
		(Water)	(Solid)
Dibenzo (a,h) anthracene		EPA	EPA 8270C/8270D/8270SIM
		8270C/8270D/8270SIM	
Dibenzofuran		EPA 8270C/8270D	EPA 8270C/8270D
1,2-Dichlorobenzene		EPA 8270C/8270D	EPA 8270C/8270D
1,3-Dichlorobenzene		EPA 8270C/8270D	EPA 8270C/8270D
1,4-Dichlorobenzene		EPA 8270C/8270D	EPA 8270C/8270D
3,3'-Dichlorobenzidine		EPA 8270C/8270D	EPA 8270C/8270D
2,4-Dichlorophenol		EPA 8270C/8270D	EPA 8270C/8270D
2,6-Dichlorophenol		EPA 8270C/8270D	EPA 8270C/8270D
Diethyl phthalate		EPA 8270C/8270D	EPA 8270C/8270D
Dimethoate		EPA 8270C/8270D	EPA 8270C/8270D
3,3-Dimethylbenzidine		EPA 8270C/8270D	EPA 8270C/8270D
p-		EPA 8270C/8270D	EPA 8270C/8270D
Dimethylaminoazobenzene			
7,12-		EPA 8270C/8270D	EPA 8270C/8270D
Dimethylbenz(a)anthracene			
Alpha-,alpha-		EPA 8270C/8270D	EPA 8270C/8270D
Dimethylphenethylamine			
2,4-Dimethylphenol		EPA 8270C/8270D	EPA 8270C/8270D
Dimethyl phthalate		EPA 8270C/8270D	EPA 8270C/8270D
Di-n-butyl phthalate		EPA 8270C/8270D	EPA 8270C/8270D
Di-n-octyl phthalate		EPA 8270C/8270D	EPA 8270C/8270D
1,3-Dinitrobenzene		EPA 8270C/8270D	EPA 8270C/8270D
1,4-Dinitrobenzene		EPA 8270C/8270D	EPA 8270C/8270D
2,4-Dinitrophenol		EPA 8270C/8270D	EPA 8270C/8270D
2,4-Dinitrotoluene		EPA 8270C/8270D	EPA 8270C/8270D
2,6-Dinitrotoluene		EPA 8270C/8270D	EPA 8270C/8270D
1,4-Dioxane		EPA 8270C/8270D	EPA 8270C/8270D
Diphenylamine		EPA 8270C/8270D	EPA 8270C/8270D
1,2-Diphenylhydrazine		EPA 8270C/8270D	EPA 8270C/8270D
Disulfoton		EPA 8270C/8270D	EPA 8270C/8270D
Diesel Range Organics		EPA 8015B/8015C, AK102,	EPA 8015B/8015C, AK102,
(DRO)		TX 1005/8015D	TX 1005/8015D
Ethyl methanesulfonate		EPA 8270C/8270D	EPA 8270C/8270D
Famphur		EPA 8270C/8270D	EPA 8270C/8270D
Fluoroanthene		EPA	EPA 8270C/8270D/8270SIM
		8270C/8270D/8270SIM	
Fluorene		EPA	EPA 8270C/8270D/8270SIM
		8270C/8270D/8270SIM	
Gasoline Range Organics		TX 1005	TX 1005
Hexachlorobenzene		EPA 8270C/8270D	EPA 8270C/8270D
Hexachlorobutadiene		EPA 8270C/8270D	EPA 8270C/8270D
Hexachlorocyclopentadiene		EPA 8270C/8270D	EPA 8270C/8270D
Hexachloroethane		EPA 8270C/8270D	EPA 8270C/8270D
Hexachloropropene		EPA 8270C/8270D	EPA 8270C/8270D
Indeno (1,2,3-cd) pyrene		EPA	EPA 8270C/8270D/8270SIM
()		8270C/8270D/8270SIM	
Isodrin		EPA 8270C/8270D	EPA 8270C/8270D
Isophorone		EPA 8270C/8270D	EPA 8270C/8270D
Isosafrole		EPA 8270C/8270D	EPA 8270C/8270D
Methapyrilene		EPA 8270C/8270D	EPA 8270C/8270D
3-Methylcholanthrene		EPA 8270C/8270D	EPA 8270C/8270D
(A2L A Cart No. 2007 01) D.		1	Page 7 of 12

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Parameter/Analyte	Non-Potable Water	Solid Hazardous Waste	Solid Hazardous Waste
		(Water)	(Solid)
2-Methyl-4,6-Dinitrophenol		EPA 8270C/8270D	EPA 8270C/8270D
Methyl methane sulfonate		EPA 8270C/8270D	EPA 8270C/8270D
2-Methylcholanthrene		EPA 8270C/8270D	EPA 8270C/8270D
1-Methylnaphthalene		EPA	EPA 8270C/8270D/8270SIM
7		8270C/8270D/8270SIM	
2-Methylnaphthalene		EPA	EPA 8270C/8270D/8270SIM
, J		8270C/8270D/8270SIM	
2-Methylphenol		EPA 8270C/8270D	EPA 8270C/8270D
3+4-Methylphenol		EPA 8270C/8270D	EPA 8270C/8270D
Naphthalene		EPA	EPA 8270C/8270D/8270SIM
Taphthalone		8270C/8270D/8270SIM	
1,4-Naphthoquinone		EPA 8270C/8270D	EPA 8270C/8270D
1-Naphthylamine		EPA 8270C/8270D	EPA 8270C/8270D
2-Naphthylamine		EPA 8270C/8270D	EPA 8270C/8270D
2-Nitroaniline		EPA 8270C/8270D	EPA 8270C/8270D
3-Nitroaniline			
4-Nitroaniline		EPA 8270C/8270D	EPA 8270C/8270D
		EPA 8270C/8270D	EPA 8270C/8270D
Nitrobenzene		EPA 8270C/8270D	EPA 8270C/8270D
2-Nitrophenol		EPA 8270C/8270D	EPA 8270C/8270D
4-Nitrophenol		EPA 8270C/8270D	EPA 8270C/8270D
Nitroquinoline-1-oxide		EPA 8270C/8270D	EPA 8270C/8270D
N-Nitrosodiethylamine		EPA 8270C/8270D	EPA 8270C/8270D
N-Nitrosodimethylamine		EPA 8270C/8270D	EPA 8270C/8270D
N-Nitrosodi-n-butylamine		EPA 8270C/8270D	EPA 8270C/8270D
N-Nitrosodi-n-propylamine		EPA 8270C/8270D	EPA 8270C/8270D
N-Nitrosodiphenylamine		EPA 8270C/8270D	EPA 8270C/8270D
N-		EPA 8270C/8270D	EPA 8270C/8270D
Nitrosomethylethylamine			
N-Nitrosomorpholine		EPA 8270C/8270D	EPA 8270C/8270D
N-Nitrosopiperidine		EPA 8270C/8270D	EPA 8270C/8270D
N-Nitrosopyrrolidine		EPA 8270C/8270D	EPA 8270C/8270D
5-Nitro-o-toluidine		EPA 8270C/8270D	EPA 8270C/8270D
2,2-oxybis(1-		EPA 8270C/8270D	EPA 8270C/8270D
chloropropane)			
Parathion, methyl		EPA 8270C/8270D	EPA 8270C/8270D
Parathion, ethyl		EPA 8270C/8270D	EPA 8270C/8270D
Pentachlorobenzene		EPA 8270C/8270D	EPA 8270C/8270D
Pentachloroethane		EPA 8270C/8270D	EPA 8270C/8270D
Pentachloronitobenzene		EPA 8270C/8270D	EPA 8270C/8270D
Pentachlorophenol		EPA	EPA
- Control of the control		8270C/8270D/8321A/8321B	8270C/8270D/8321A/8321B
Phenacetin		EPA 8270C/8270D	EPA 8270C/8270D
Phenanthrene		EPA	EPA 8270C/8270D/8270SIM
Thenanthene		8270C/8270D/8270SIM	El 11 02/0C/02/0D/02/05IVI
Phenol		EPA 8270C/8270D	EPA 8270C/8270D
1,4-Phenylenediamine		EPA 8270C/8270D EPA 8270C/8270D	EPA 8270C/8270D
Phorate		EPA 8270C/8270D EPA 8270C/8270D	EPA 8270C/8270D
2-Picoline		EPA 8270C/8270D	EPA 8270C/8270D
Pronamide		EPA 8270C/8270D	EPA 8270C/8270D
Pyrene		EPA	EPA 8270C/8270D/8270SIM
D ' ' L'		8270C/8270D/8270SIM	EDA 02700/02705
Pyridine		EPA 8270C/8270D	EPA 8270C/8270D
	evised 10/12/2011	1/1 //	Page 8 of 13

Parameter/Analyte	Non-Potable Water	Solid Hazardous Waste	Solid Hazardous Waste
		(Water)	(Solid)
Safrole		EPA 8270C/8270D	EPA 8270C/8270D
Sulfotepp		EPA 8270C/8270D	EPA 8270C/8270D
1,2,4,5-Tetrachlorobenzene		EPA 8270C/8270D	EPA 8270C/8270D
2,3,4,6-Tetrachlorophenol		EPA 8270C/8270D	EPA 8270C/8270D
Thionazin		EPA 8270C/8270D	EPA 8270C/8270D
o-Toluidine		EPA 8270C/8270D	EPA 8270C/8270D
1,2,4-Trichlorobenzene		EPA 8270C/8270D	EPA 8270C/8270D
2,4,5-Trichlorophenol		EPA 8270C/8270D	EPA 8270C/8270D
2,4,6-Trichlorophenol		EPA 8270C/8270D	EPA 8270C/8270D
o,o,o-Triethyl		EPA 8270C/8270D	EPA 8270C/8270D
phosphorothioate			2111 027 007 027 02
1,3,5-Trinitrobenzene		EPA 8270C/8270D	EPA 8270C/8270D
Tris(2,3-Dibromopropyl)		EPA 8270C/8270D	EPA 8270C/8270D
phosphate		E171 0270C/0270B	E171 0270 C/0270B
Motor Oil (Residual Range		EPA 8015B/8015C, AK103	EPA 8015B/8015C, AK103
Organics)		E171 0013B/0013C, 7111103	E171 0013B/0013C, 711103
Organies)			
Pesticides/Herbicides/PCBs			
Aldrin		EPA 8081A/8081B	EPA 8081A/8081B
Atrazine		EPA 8141A/8141B	EPA 8141A/8141B
Azinophos ethyl		EPA 8141A/8141B	EPA 8141A/8141B
Azinophos methyl		EPA 8141A/8141B	EPA 8141A/8141B
alpha-BHC		EPA 8081A/8081B	EPA 8081A/8081B
Beta-BHC		EPA 8081A/8081B	EPA 8081A/8081B
delta-BHC		EPA 8081A/8081B	EPA 8081A/8081B
Gamma-BHC		EPA 8081A/8081B	EPA 8081A/8081B
Bolstar		EPA 8141A/8141B	EPA 8141A/8141B
Alpha-Chlordane		EPA 8081A/8081B	EPA 8081A/8081B
Gamma-Chlordane		EPA 8081A/8081B	EPA 8081A/8081B
Chlordane (technical)		EPA 8081A/8081B	EPA 8081A/8081B
Chloropyrifos		EPA 6061A/6061B	EPA 6061A/6061B
Chloropythos		8081A/8081B/8141A/8141B	8081A/8081B/8141A/8141B
Coumaphos		EPA 8141A/8141B	EPA 8141A/8141B
2,4-D		EPA 8151A/8321A	EPA 8151A/8321A
Dalapon		EPA 8151A/8321A EPA 8151A/8321A	EPA 8151A/8321A EPA 8151A/8321A
•			
2,4-DB		EPA 8151A/8321A	EPA 8151A/8321A
4,4'-DDD		EPA 8081A/8081B	EPA 8081A/8081B
4,4'-DDE		EPA 8081A/8081B	EPA 8081A/8081B
4,4',-DDT		EPA 8081A/8081B	EPA 8081A/8081B
Demeton-O		EPA 8141A/8141B	EPA 8141A/8141B
Demeton-S		EPA 8141A/8141B	EPA 8141A/8141B
Demeton, total		EPA 8141A/8141B	EPA 8141A/8141B
Diazinon		EPA 8141A/8141B	EPA 8141A/8141B
Dicamba		EPA 8151A/8321A	EPA 8151A/8321A
Dichlorovos		EPA 8141A/8141B	EPA 8141A/8141B
Dichloroprop		EPA 8151A/8321A	EPA 8151A/8321A
Dicofol		EPA 8081A/8081B	EPA 8081A/8081B
Dieldrin		EPA 8081A/8081B	EPA 8081A/8081B
Dimethoate		EPA 8141A/8141B	EPA 8141A/8141B
Dinoseb		EPA 8151A/8321A	EPA 8151A/8321A
Disulfoton		EPA 8141A/8141B	EPA 8141A/8141B
Endosulfan I		EPA 8081A/8081B)	EPA 8081A/8081B

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Parameter/Analyte	Non-Potable Water	Solid Hazardous Waste	Solid Hazardous Waste
		(Water)	(Solid)
Endosulfan II		EPA 8081A/8081B	EPA 8081A/8081B
Endonsulfan sulfate		EPA 8081A/8081B	EPA 8081A/8081B
Endrin		EPA 8081A/8081B	EPA 8081A/8081B
Endrin aldehyde		EPA 8081A/8081B	EPA 8081A/8081B
Endrin ketone		EPA 8081A/8081B	EPA 8081A/8081B
EPN		EPA 8141A/8141B	EPA 8141A/8141B
Ethoprop		EPA 8141A/8141B	EPA 8141A/8141B
Ethyl parathion		EPA 8141A/8141B	EPA 8141A/8141B
Famphur		EPA 8141A/8141B	EPA 8141A/8141B
Fensulfothion		EPA 8141A/8141B	EPA 8141A/8141B
Fenthion		EPA 8141A/8141B	EPA 8141A/8141B
Heptachlor		EPA 8081A/8081B	EPA 8081A/8081B
Heptachlor epoxide	<u> </u>	EPA 8081A/8081B	EPA 8081A/8081B
Hexachlorobenzene		EPA 8081A/8081B	EPA 8081A/8081B
Malathion		EPA 8141A/8141B	EPA 8141A/8141B
MCPA		EPA 8151A/8321A	EPA 8151A/8321A
MCPP		EPA 8151A/8321A EPA 8151A/8321A	EPA 8151A/8321A
Merphos		EPA 8141A/8141B	EPA 8141A/8141B
Methoxychlor		EPA 8081A/8081B	EPA 8081A/8081B
Methyl parathion		EPA 8081A/8081B EPA 8141A/8141B	EPA 8141A/8141B
Mevinphos		EPA 8141A/8141B	EPA 8141A/8141B
Naled		EPA 8141A/8141B	EPA 8141A/8141B
PCB-1016 (Arochlor)		EPA 8082/8082A	EPA 8082/8082A
PCB-1221		EPA 8082/8082A	EPA 8082/8082A
PCB-1232		EPA 8082/8082A	EPA 8082/8082A
PCB-1242		EPA 8082/8082A	EPA 8082/8082A
PCB-1248		EPA 8082/8082A	EPA 8082/8082A
PCB-1254		EPA 8082/8082A	EPA 8082/8082A
PCB-1260		EPA 8082/8082A	EPA 8082/8082A
PCB-1262		EPA 8082/8082A	EPA 8082/8082A
PCB-1268		EPA 8082/8082A	EPA 8082/8082A
Phorate		EPA 8141A/8141B	EPA 8141A/8141B
Phosmet		EPA 8141A/8141B	EPA 8141A/8141B
Propazine		EPA 8141A/8141B	EPA 8141A/8141B
Ronnel		EPA 8141A/8141B	EPA 8141A/8141B
Simazine		EPA	EPA
		8081A/8081B/8141A/8141B	8081A/8081B/8141A/8141B
Stirophos		EPA 8141A/8141B	EPA 8141A/8141B
Sulfotepp		EPA 8141A/8141B	EPA 8141A/8141B
2,4,5-T		EPA 8151A/8321A	EPA 8151A/8321A
Thionazin		EPA 8141A/8141B	EPA 8141A/8141B
Tokuthion		EPA 8141A/8141B	EPA 8141A/8141B
2,4,5-TP		EPA 8151A/8321A	EPA 8151A/8321A
Toxaphene		EPA 8081A/8081B	EPA 8081A/8081B
Trichloronate		EPA 8141A/8141B	EPA 8141A/8141B
o,o,o-triethylphos		EPA 8141A/8141B	EPA 8141A/8141B
phorothioate			
Explosives			
1,3,5-Trinitrobenzene		EPA	EPA
		8330A/8330B/8321A/8321B	8330A/8330B/8321A/8321B

Parameter/Analyte	Non-Potable Water	Solid Hazardous Waste	Solid Hazardous Waste
1 arameter/Anaryte	1von-1 otable water	(Water)	(Solid)
1,3-Dinitrobenzene		EPA	EPA
1,5-Dimitiobelizelle		8330A/8330B/8321A/8321B	8330A/8330B/8321A/8321B
2,4,6-Trinitrotoluene		EPA	EPA
2,4,0-1111111010101011		8330A/8330B/8321A/8321B	8330A/8330B/8321A/8321B
3,5-Dinitroaniline		8330B	8330B
2,4-Dinitrotoluene		EPA	EPA
2,4-Dinitrototuene			
2 (Division 1) - 1		8330A/8330B/8321A/8321B	8330A/8330B/8321A/8321B
2,6-Dinitroltoluene		EPA 8330A/8330B/8321A/8321B	EPA 8330A/8330B/8321A/8321B
2-Amino-4,6-dinitrotoluene		EPA	EPA
2-Ammo-4,0-ammrotoruene			8330A/8330B/8321A/8321B
2 Ni (ma) - 1		8330A/8330B/8321A/8321B	
2-Nitrotoluene		EPA	EPA
2 77 1		8330A/8330B/8321A/8321B	8330A/8330B/8321A/8321B
3-Nitrotoluene		EPA	EPA
		8330A/8330B/8321A/8321B	8330A/8330B/8321A/8321B
4-Amino-2,6-dinitrotoluene		EPA	EPA
1.27		8330A/8330B/8321A/8321B	8330A/8330B/8321A/8321B
4-Nitrotoluene		EPA	EPA
		8330A/8330B/8321A/8321B	8330A/8330B/8321A/8321B
Nitrobenzene		EPA	EPA
		8330A/8330B/8321A/8321B	8330A/8330B/8321A/8321B
Nitroglycerin		EPA	EPA
		8330A/8330B/8321A/8321B	8330A/8330B/8321A/8321B
Octahydro-1,3,5,7-		EPA	EPA
tetrabitro-1,3,5,7-		8330A/8330B/8321A/8321B	8330A/8330B/8321A/8321B
tetrazocine (HMX)			
Pentaerythritoltetranitrate		EPA	EPA
(PETN)		8330A/8330B/8321A/8321B	8330A/8330B/8321A/8321B
Picric acid		EPA 8330A/8330B	EPA 8330A/8330B
RDX (hexahydro-1,3,5-		EPA	EPA
trinitro-1,3,5-triazine)		8330A/8330B/8321A/8321B	8330A/8330B/8321A/8321B
Tetryl (methyl2,4,6-		EPA	EPA
trinitrophenylnitramine		8330A/8330B/8321A/8321B	8330A/8330B/8321A/8321B
<u>Hydrazines</u>			
Hydrazine	SOP DV WC-0077	SOP DV WC-0077	SOP DV WC-0077
Monomethyl hydrazine	SOP DV WC-0077	SOP DV WC-0077	SOP DV WC-0077
1,1-Dimethylhydrazine	SOP DV WC-0077	SOP DV WC-0077	SOP DV WC-0077
Perfluorinated			
Hydrocarbons (PFCs) and			
Perfluorinated Sulfonates			
(PFSs)			
Perfluorobutanoic acid	SOP DV-LC-0012	SOP DV-LC-0012	SOP DV-LC-0012
Perfluoropentanoic acid	SOP DV-LC-0012	SOP DV-LC-0012	SOP DV-LC-0012
Perfluorohexanoic acid	SOP DV-LC-0012	SOP DV-LC-0012	SOP DV-LC-0012
Perfluoroheptanoic acid	SOP DV-LC-0012	SOP DV-LC-0012	SOP DV-LC-0012
Perfluorooctanoic acid	SOP DV-LC-0012	SOP DV-LC-0012	SOP DV-LC-0012
Perfluorononanoic acid	SOP DV-LC-0012	SOP DV-LC-0012	SOP DV-LC-0012
Perfluorodecanoic acid	SOP DV-LC-0012	SOP DV-LC-0012	SOP DV-LC-0012
Perfluoroundecanoic acid	SOP DV-LC-0012	SOP DV-LC-0012	SOP DV-LC-0012
Perfluorododecanoic acid	SOP DV-LC-0012	SOP DV-LC-0012	SOP DV-LC-0012
1 chiuorododecanoic acid	501 D V-LC-0012	SOF DV-LC-0012	501 D V-LC-0012

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Parameter/Analyte	Non-Potable Water	Solid Hazardous Waste	Solid Hazardous Waste
		(Water)	(Solid)
Perfluorotridecanoic acid	SOP DV-LC-0012	SOP DV-LC-0012	SOP DV-LC-0012
Perfluorotetradecanoic acid	SOP DV-LC-0012	SOP DV-LC-0012	SOP DV-LC-0012
Perfluorobutane Sulfonate	SOP DV-LC-0012	SOP DV-LC-0012	SOP DV-LC-0012
Perfluorohexane Sulfonate	SOP DV-LC-0012	SOP DV-LC-0012	SOP DV-LC-0012
Perfluorooctane Sulfonate	SOP DV-LC-0012	SOP DV-LC-0012	SOP DV-LC-0012
Perfluorodecane Sulfonate	SOP DV-LC-0012	SOP DV-LC-0012	SOP DV-LC-0012
Perfluorooctane	SOP DV-LC-0012	SOP DV-LC-0012	SOP DV-LC-0012
Sulfonamide			
N-Nitrosodimethylamine	SOP DV-LC-0019	SOP DV-LC-0019	SOP DV-LC-0019
(NDMA)			
Hazardous Waste			
Characteristics			
Conductivity		EPA 9050A	EPA 9050A
Corrosivity		EPA 9040B	9045C
Ignitibility	EPA 1010/EPA 1010A	EPA 1010/EPA 1010A	EPA 1010/EPA 1010A
Paint Filter Liquids Test		EPA 9095A	EPA 9095A
Synthetic Precipitation		EPA 1312	EPA 1312
Leaching Procedure (SPLP)		2111 1312	
ToxicityCharacteristic		EPA 1311	EPA 1311
Leaching Procedure			
Organic Prep Methods			
Separatory Funnel Liquid-		EPA 3510C	
Liquid Extraction		217133100	
Continuous Liquid-Liquid		EPA 3520C	
Extraction Extraction		217133200	
Soxhlet Extraction			EPA 3540C
Microwave Extraction			EPA 3546
Ultrasonic Extraction			EPA 3550B
Ultrasonic Extraction			EPA 3550C
Waste Dilution		EPA 3580A	EPA 3580A
Solid Phase Extraction		EPA 3535A	EPA 5030B
Volatiles Purge and trap		EPA 5030B	EPA 5035
Volatiles purge and trap for		LI A 3030B	LI A 3033
soils			
30113			
Organic Cleanup			
Procedures			
Florisil Cleanup		EPA 3620B	EPA 3620B
Florisil Cleanup		EPA 3620C	EPA 3620C
Sulfur Cleanup		EPA 3660B	EPA 3660B
Sulfuric		EPA 3665A	EPA 3665A
Acid/Permanganate		DIA 3003A	LITI SOUST
Cleanup			
Cicanup			
Metals Digestion			
Acid Digestion Total		EPA 3005A	<u> </u>
Recoverable or Dissolved		DIA 3003A	
Metals			
Acid Digestion for Total		EPA 3010A	<u> </u>
Metals		LI A JUIUA	
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Parameter/Analyte	Non-Potable Water	Solid Hazardous Waste	Solid Hazardous Waste
		(Water)	(Solid)
Acid Digestion for Total		EPA 3020A	
Metals			
Acid Digestion of			EPA 3050B
Sediments, Sludges and			
Soils			

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Presented this 5th day of October 2011.

President & CEO

For the Accreditation Council Certificate Number 2907.01

Valid to October 31, 2013

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Title: Polynuclear Aromatic Hydrocarbons by GC/MS Selected Ion Monitoring (SIM) [SW 846 Method 8270C and 8270D]

Approvals (Signature/Date):							
Joe Kempton Date Technical Specialist John P. Morris Date Quality Assurance Manager	Adam Alban Date Health & Safety Manager / Coordinator Love L Chamiel F/3//2						

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1.0 Scope and Application

1.1 This procedure is a Gas Chromatography/Mass Spectrometry (GC/MS) technique for the analysis of polynuclear aromatic hydrocarbons (PAH) and heterocyclic compounds at the part per trillion (ng/L or ng/kg) level in waters or solids. This procedure follows the general guidelines of EPA Methods 8270C and 8270D for Selected Ion Monitoring (SIM) analysis.

- 1.2 The SIM technique optimizes quantitative information at the expense of qualitative information gained from other methods of analysis. It is important to note that this procedure is intended for the analysis of samples previously characterized by another method such as open-scan 8270C/D. The initial characterization is necessary to avoid misidentification of the parent compounds producing the ions used for this analysis.
- 1.3 In addition, this procedure is appropriate only for sample analytes of interest at less than 10,000 ng/L or 330,000 ng/kg. Samples containing semivolatile organics at concentrations greater than 10,000 ng/L and 330,000 ng/kg should be analyzed by a method designed to detect at higher (part per billion) levels. Samples at these levels may still be analyzed by this procedure, however, extra measurement uncertainty would be introduced because of the sample dilutions that would be required.
- 1.4 This procedure is applicable to water and soil samples. For water samples, 1 liter of water is extracted. A 4-liter extraction procedure to achieve lower detection limits is described in SOP DV-MS-0005. For soil samples, a sample aliquot of 30 g is extracted.

1.5 Analytes, Matrix(s), and Reporting Limits

The standard list of compounds that can be analyzed by this procedure is shown in Table IV. Typical reporting limits are 100 ng/L for aqueous samples and 5.0 ug/Kg for soil samples for the PAH compounds.

2.0 Summary of Method

2.1 Sample Preparation

2.1.1 Aqueous Samples

Analytes of interest are extracted from water samples using separatory funnel extraction (EPA 3510C) described in SOP DV-OP-0006. Samples are prepared by the continuous-liquid-liquid-extraction (CLLE) technique (EPA 3520C), which is covered in SOP DV-OP-0008. The PAH compounds are extracted from the sample without any adjustment to pH. The concentration of organic extracts is covered in SOP DV-OP-0007.

2.1.2 Solid Samples

Solid samples are extracted by sonication (EPA 3550C), which is covered in SOP DV-OP-0016 or by microwave extraction (EPA 3546) described is SOP DV-OP-0015. The extraction solvent is a 1:1 mixture of methylene chloride and acetone.

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2.2 Instrumental Analysis

Quantitation of the extracted compounds is performed by gas chromatography - mass spectrometry (GC/MS) in the selected ion monitoring mode (SIM). Routine instrument conditions and the ions used for analysis are shown in Tables I and IV, respectively.

Development of a successful SIM method requires identifying the ions to be monitored, the ion dwell times, the ions in each group, and the timing for switching between groups. A quantitation ion is selected with a confirmation ion being monitored for identification purposes (see Table IV). Switching times are set where there is adequate resolution (a gap of 1-2 minutes) between peaks. If there is inadequate time between eluting peaks, small retention time shifts may cause peaks to partially or completely disappear as there are changes in the ions monitored. Dwell times will be set by default once the ions per group and the switching times are identified in the method. These can be adjusted manually in order to optimize sensitivity as needed.

3.0 <u>Definitions</u>

- 3.1 Refer to TestAmerica Denver's Quality Assurance Manual (QAM) and SOP DV-QA-003P for definitions of the quality control terms used in this document.
- 3.2 <u>Selected Ion Monitoring</u> A mass spectrometry technique that provides lower detection level capability by monitoring fewer mass scans for longer periods of time than is done in open-scan methods.
- **3.3** Primary Ion Area The signal chosen for quantitation purposes.
- **3.4** Secondary Ion Area The signal chosen for identification and confirmation purposes.

4.0 <u>Interferences</u>

- 4.1 Method interferences may be caused by contaminants in solvents, reagents, glassware, and other sample processing hardware that lead to discrete artifacts and/or elevated baselines in the ion current profiles. All of these materials must be routinely demonstrated to be free from interferences under the conditions of the analysis by running laboratory reagent blanks. The use of high purity reagents and solvents helps to minimize interference problems.
- **4.2** Matrix interferences may be caused by contaminants that are co-extracted from the sample. The extent of matrix interferences will vary considerably from source to source, depending upon the nature of the environment being sampled.
- 4.3 An interference that is unique to selected ion monitoring techniques can arise from the presence of an interfering compound which produces the same ion used for quantitation of one of the PAHs. This event results in a positive interference to the reported value for the compound of interest. This interference is controlled to some degree by acquiring data for a confirmation ion. If the ion ratios between the quantitation ion and the confirmation ion are not within the specified limits, then interferences may be present. Open scan analysis to identify compounds throughout the mass range is the most reliable assurance against reporting false positives.

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5.0 Safety

Employees must abide by the policies and procedures in the Environmental Health and Safety Manual, Radiation Safety Manual and this document.

This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, nitrile or latex gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1 Specific Safety Concerns or Requirements

- 5.1.1 Eye protection that satisfies ANSI Z87.1, laboratory coat, and nitrile gloves must be worn while handling samples, standards, solvents, and reagents. Disposable gloves that have been contaminated must be removed and discarded; non-disposable gloves must be cleaned immediately. Latex and vinyl gloves provide no protection against the organic solvents used in this method. Nitrile or similar gloves must be used.
- 5.1.2 The gas chromatograph and mass spectrometer contain zones that have elevated temperatures. The analyst needs to be aware of the locations of those zones, and must cool them to room temperature prior to working on them.
- **5.1.3** The mass spectrometer is under deep vacuum. The mass spectrometer must be brought to atmospheric pressure prior to working on the source.
- 5.1.4 There are areas of high voltage in both the gas chromatograph and the mass spectrometer. Depending on the type of work involved, either turn the power to the instrument off, or disconnect the instrument from its source of power.

5.2 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating.

NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.

A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

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Materials with Serious or Significant Hazard Rating

Material ⁽¹⁾	Hazards	Exposure Limit ⁽²⁾	Signs and symptoms of exposure
Methanol	Flammable Poison Irritant	200 ppm - TWA	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.
Methylene Chloride	Carcinogen Irritant	25 ppm - TWA 125 ppm - STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.
PAH standards can contain all or some of the following: benzo(a)anthracene benzo(b)fluoranthene benzo(k)fluoranthene benzo(a)pyrene chrysene dibenz(a,h)anthracene indeno(1,2,3-cd)pyrene	Carcinogen Carcinogen Carcinogen Carcinogen Carcinogen Carcinogen Carcinogen	0.2 mg/m ³ - PEL	Standards contain low concentrations of compounds known to be or suspected to be carcinogens. All PAH compounds are considered to be hazardous, toxic, and irritants. Some or all are reported human carcinogens, mutagens, and/or teratogens.
naphthalene		10 ppm - PEL	

⁽¹⁾ Always add acid to water to prevent violent reactions.

6.0 Equipment and Supplies

6.1 <u>Instrumentation</u>

Gas Chromatograph (See Table I for operating conditions)

6.1.1 The analytical system includes a temperature programmable gas chromatograph and all required accessories including syringes, analytical columns, and gases. The injection port is designed for on-column injection when using packed columns and for splitless injection when using capillary columns.

⁽²⁾ Exposure limit refers to the OSHA regulatory exposure limit.

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6.1.2 Mass Spectrometer (See Table I for operating conditions)

A mass spectrometer operating at 70 eV (nominal) electron energy in the electron impact ionization mode and tuned to maximize the sensitivity of the instrument to the compounds being analyzed. The GC capillary column is fed directly into the ion source of the mass spectrometer.

- 6.1.3 A computer system interfaced to the mass spectrometer that allows the continuous acquisition and storage on machine-readable media of all mass spectra obtained throughout the duration of the chromatographic program. The computer has software that allows searching any GC/MS data file for ions of a specific mass and plotting such ion abundances versus time or scan number. The computer allows acquisition at pre-selected mass windows for selected ion monitoring.
- 6.1.4 Please refer to the *Master list of Documents, Software, and Hardware* located on G:\QA\Read\Master List of Documents for the current software and hardware to be used for data processing.

6.2 Supplies

- All glassware used, both within the scope of this SOP and for the initial sample extraction (see SOPs DV-OP-0006, DV-OP-0008, DV-OP-0007, DV-OP-0015, and DV-OP-0016) must be scrupulously cleaned. Clean all glassware as soon as possible after use by rinsing with the last solvent used in it. This should be followed by detergent washing with hot water, and rinses with tap water, reagent water, and finally with acetone.
- 6.2.2 Glassware should <u>not</u> be oven dried or heated in a muffle furnace. Successive solvent rinses of the CLLE, separatory funnel, sonication, and Kuderna-Danish glassware are required to minimize low level contamination of samples.
- **6.2.3** Store glassware inverted or in sealed containers capped with aluminum foil.
- **6.2.4** Gas-tight syringes, various sizes, and SMI pipettors.
- **6.2.5** Serological pipettes are used for final extract volume measurement.
- **6.2.6** Micro reaction vessels, 1.8 mL vials with Teflon caps, for storing concentrated extracts.
- **6.2.7** Column A Varian VF-5MS 30-meter fused silica capillary column, 0.5 μm film thickness, 0.25mm ID, plus 10-meter EZguard, or equivalent.

7.0 Reagents and Standards

7.1 Reagents

All solvents are reagent grade or higher unless specified otherwise. See SOPs CA-Q-S-001 and CA-Q-S-001 DV-1 for a description of the program for testing solvents prior to use.

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- **7.1.1** Methanol, reagent grade.
- **7.1.2** Methylene chloride, reagent grade.
- **7.1.3** Helium gas, 99% + purity.

7.2 Standards

Commercial standards are received in flame-sealed ampoules or neat, 100% concentration solutions. Standards are verified before use. Details concerning verification of standards are given in SOP DV-QA-0015. Stock standards are stored refrigerated at \leq 6 °C. All stock standards must be protected from light. Stock standards are monitored for signs of degradation or evaporation. The standards must be replaced annually from the date of receipt or earlier, if the vendor indicates an earlier date.

7.2.1 GC/MS Tuning Standard

A methylene chloride solution containing decafluorotriphenylphosphine (DFTPP) at a concentration of $50 \mu g/mL$ is prepared.

7.2.2 Calibration Standards

Calibration standards for the initial calibration (ICAL) are prepared at 7 concentrations to cover the calibration range by diluting vendor stock standard solutions using methylene chloride. The standards are prepared directly in autosampler vials by using a calibrated microliter syringe to deliver the appropriate volumes of stock standard solution, internal standard solution, and methylene chloride. The following table summarizes a typical set of calibration standards prepared using a PAH SIM stock standard with a concentration of 20 μ g/mL for levels 4 through 7. A secondary PAH SIM stock standard with a concentration of 2 μ g/mL is used to prepare levels 1 through 3:

Vol of Stock Used (μL)	Methylene Chloride Added (μL)	Internal Standard Added (μL)	Final Volume (μL)	Final Conc of PAH (μg/mL)
5	495	50	500	0.02
25	475	50	500	0.1
75	425	50	500	0.3
15	485	50	500	0.6
30	470	50	500	1.2
62.5	437.5	50	500	2.5
125	375	50	500	5.0

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7.2.3 Initial Calibration Verification (ICV) Standard

A second source initial calibration verification (ICV) standard is prepared using a standard solution that is obtained from a source independent from the source that supplies the standard used for the initial calibration. The final PAH SIM concentration for this ICV standard is 1.2 μ g/mL.

7.2.4 Continuing Calibration Verification (CCV) Standard

A standard with the same analytes, concentrations, and lot numbers as the 600 ng/mL calibration standard. The standard may be from the same preparation as the initial calibration or prepared at a later date.

7.2.5 Surrogate Spiking Solutions

The surrogate spike solution contains neutral surrogates at concentrations of 500 ng/mL in methanol. Table II lists the surrogate compounds for the standard list of PAHs. One (1.0) milliliter of the surrogate spike solution is added to a 1.0-liter aliquot of an aqueous sample, or a 30-gram aliquot of a soil or solid sample.

7.2.6 Internal Standard (IS) Solutions

A solution containing each of the internal standards at a concentration of 600 ng/mL each in methylene chloride is prepared from vendor stock. Table III lists the IS compounds.

7.2.7 LCS, MS, and MSD Spike Solution

A methanol solution containing the requested spike compounds at the concentration of 900 ng/mL each for PAH compound is prepared from vendor stock solutions. Following are the final sample concentrations of the spiked compounds for the water and solid extractions:

PAH in water: 900 ng/L

PAH in soil matrices: 30.0 μg/kg

7.3 All stock and working standards are stored according to the manufacturer's instructions. Dilutions from stocks may not be assigned expiration dates that exceed the stock standard expiration date set by the manufacturer.

8.0 Sample Collection, Preservation, Shipment and Storage

8.1 Sample Amounts

8.1.1 Water samples are collected in pre-cleaned amber glass bottles fitted with a Teflon-lined cap. To guarantee the ability to meet routine reporting limits, two full one-liter bottles of sample should be provided. Additional one-liter portions are needed to satisfy the requirements for matrix spikes and duplicate matrix spikes.

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8.1.2 Soil samples are collected in an 8-ounce, pre-cleaned, wide-mouth jar with a Teflon-lined lid.

- 8.1.3 If insufficient sample is provided to perform the matrix spikes described in Section 9.4, analysts must prepare a Nonconformance Memo (NCM) as required by SOP DV-QA-0031. The NCM is routed to the laboratory project manager so that the problem can be discussed with the client.
- 8.2 Samples are chilled to a temperature between 0 and 6 °C immediately after collection and shipped via overnight carrier to the laboratory.
- 8.3 Samples and excess sample volume must be stored refrigerated at ≤ 6 °C from when the log-in process is completed (see SOP DV-QA-0003) to storage after analysis.
- 8.4 Water samples must be extracted within 7 days of the time of sample collection, while solid samples must be extracted within 14 days of sampling. Extracts must be analyzed within 40 days from sample extraction.

9.0 Quality Control

- **9.1** The minimum quality controls (QC), acceptance criteria and corrective actions are described in this section. When processing samples in the laboratory, use the LIMS Method Comments to determine specific QC requirements that apply.
 - **9.1.1** The laboratory's standard QC requirements, the process of establishing control limits, and the use of control charts are described more completely in TestAmerica Denver policy DV-QA-003P, *Quality Assurance Program*.
 - 9.1.2 Specific QC requirements for Federal programs, e.g., Department of Defense (DoD), Department of Energy (DOE), AFCEE, etc., are described in TestAmerica Denver policy DV-QA-024P, *Requirements for Federal Programs*.
 - 9.1.3 Project-specific requirements can override the requirements presented in this section when there is a written agreement between the laboratory and the client, and the source of those requirements should be described in the project documents. Project specific requirements are communicated to the analyst via Method Comments and special instructions in the LIMS and in the Quality Assurance Summaries (QAS) available in the public folders.
 - 9.1.4 Any QC result that fails to meet control criteria must be documented in a Nonconformance Memo (NCM). The NCM is automatically sent to the laboratory supervisor and Project Manager by e-mail so that the client can be notified as appropriate. The NCM process is described in more detail in SOP DV-QA-0031. This is in addition to the corrective actions described in the following sections.

9.2 Method Blank (MB)

A method blank is processed and analyzed with each analytical batch, not to exceed 20 samples. For aqueous samples, the method blank consists of reagent water spiked with surrogates. For soil samples, the method blank is Ottawa sand spiked with surrogates. This sand is mixed with sodium sulfate for extraction by ultrasonication. Method blanks

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are used to assess whether the laboratory has contributed contamination to the sample analysis process that adversely affects the accuracy of the determination of target analytes. The goal is to have no detectable contaminants in the method blank. However, due to the sensitivity of this analysis, it is not uncommon to detect target analytes at levels above the method detection limit (MDL).

Acceptance Criteria: MB results must be less than ½ the reporting limit.

Corrective Action: If the MB exceeds ½ the RL for any target analyte, then one of the

following must apply for acceptance of the batch:

The blank contamination is less than $^{1}/_{10}$ of the measured concentration of any sample in the associated preparation batch,

The blank contamination is less than the concentration present in

the samples and is less than $\frac{1}{10}$ of the regulatory limit, or

The same contaminants are <u>not</u> found in the associated samples.

NOTE: Positive method blank results below the reporting limit should still

be evaluated by the analyst for potential impact on sample results

at or near the reporting limit.

9.3 Laboratory Control Samples (LCS)

A Laboratory Control Sample (LCS) is processed and analyzed with each analytical batch not to exceed 20 samples. For aqueous samples, the LCS consists of reagent water spiked with the analytes of interest and surrogates. For soil samples, the LCS is Ottawa sand spiked with analytes of interest and surrogates. For ultrasonic extraction, sodium sulfate is added to the reagent sand. The LCS spiking solution is described in Section 7.2.7. LCS results are used to determine whether the analytical system is in control. Depending on project requirements, a duplicate LCS (LCSD) may be required to assess the precision of the analytical system.

Acceptance Criteria: The percent recovery for each requested target analyte in the LCS must fall within the established control limits (found in the LIMS system). If the percent recovery for an analyte that is in the LCS, but that is not a requested target analyte for the associated samples, does not fall within the control limits, but all of the requested target analytes that are in the LCS do fall within the control limits, then the LCS is considered to have met acceptance criteria.

Corrective Action:

If the percent recovery for any analyte in the LCS exceeds the upper control limit and the analyte is not detected in any of the associated samples, then no further action is required, and data are reported with discussion in the case narrative.

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If the percent recovery for any analyte in the LCS exceeds the upper control limit and the analyte is detected in any of the associated samples, then reanalyze the LCS. If similar results are obtained on the second attempt, then investigate and correct any problems. Re-extract and reanalyze the analytical batch.

If the percent recovery for any analyte in the LCS is below the lower control limit, reanalyze the LCS. If similar results are obtained on the second attempt, then investigate and correct any problems. Re-extract and reanalyze the analytical batch.

If re-extraction of samples is not possible, qualify data and explain in the case narrative.

Document all corrective actions taken in an NCM and in the case narrative.

9.4 Matrix Spike and Spike Duplicate (MS/MSD)

One matrix spike (MS) sample and one matrix spike duplicate (MSD) sample are prepared and analyzed for each analytical batch not to exceed 20 samples. An MS sample is a field sample to which known amounts of the target analytes, as well as the surrogates, have been added. An MSD is a second aliquot of the same sample that is spiked the same as the MS. The MS/MSD spiking solution is described in Section 7.2.7. MS results are used to assess the effects of the sample matrix on the accuracy of the analytical system. The MSD results are used to assess the effects of the sample matrix on the precision of the analytical system. Given the expected variability in sample matrix, the MS/MSD results are applicable to only the sample used to prepare the MS and MSD. MS/MSD results should not be extrapolated to other samples without extensive investigation and characterization to demonstrate similarity between samples.

Acceptance Criteria: The percent recovery for each requested target analyte in the MS and MSD must fall within the established control limits (found in the LIMS system). The relative percent difference (RPD) between the MS and MSD must be less than or equal to the established control limit.

Corrective Action:

If the percent recovery for any requested target analyte in the MS or MSD falls outside of the established control limits, or the RPD between the MS and MSD exceeds the established limit, but the LCS and instrument QC are within control limits, then the analytical system is considered to be in control. Review sample preparation for any problems and if none are identified, then the MS/MSD failure(s) may be attributed to matrix effects, and data may be reported with an explanation in the case narrative. Depending on project requirements, it may be necessary to reanalyze the MS/MSD to confirm matrix effects.

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9.5 Internal Standards

The internal standards listed in Table III and described in Section 7.2.6 are spiked at the same level in all field sample extracts, QC sample extracts, instrument blanks, and calibration standards.

Acceptance Criteria: The peak area for each internal standard in each field sample and

QC sample extract should be between 50% and 200% of the peak area for the same internal standard in the midlevel standard of the

initial calibration.

Corrective Action: If the internal standard fails acceptance criteria, then perform the

following corrective actions:

Inspect system for malfunction and correct as needed.

Reanalyze the affected samples.

If the interference cannot be corrected for field samples, the earlier analysis is reported with discussion in the case narrative.

If QC samples have internal standard failures that are confirmed by re-analysis, the cause of the failures must be investigated.

All corrective actions must be documented.

9.6 Surrogate Compound Analysis

Surrogate compounds listed in Table II and described in Section 7.2.4 are added to all field and QC samples prior to extraction. Surrogate recoveries are used to assess individual sample matrix effects on sample preparation and analysis.

Acceptance Criteria: Surrogate recoveries must fall within established control limits.

QC sample results are not acceptable unless the surrogate

recoveries for those samples are in control.

Corrective Action: Corrective action must be considered for any surrogate failure

and may depend on project-specific instructions. Lacking instructions to the contrary the following actions shall be taken:

Evaluate sample chromatogram and other QC.

If the surrogate(s) fail in the LCS and/or method blank, then reprepare and reanalyze all associated samples.

For surrogate failures in field samples, re-prepare and reanalyze the samples, unless matrix interference is evident from earlier analysis or from chromatograms. In the latter case, contact the client and the matrix interference in the case narrative.)

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9.7 Instrument QC

9.7.1 Instrument Optimization

- 9.7.1.1 The GC/MS system must be tuned to meet manufacturer's specifications, using a suitable calibration such as perfluorotrinbutylamine (FC-43). This is performed through the auto-tune feature in the software. The mass calibration and resolution of the GCMS system is then verified by the analysis of DFTPP prior to the analysis of any standards or samples.
- 9.7.1.2 The instrument is tuned for DFTPP (decafluorotriphenylphosphine), calibrated initially with a seven-point calibration curve, and verified each 12-hour shift that samples are to be run with one or more continuing calibration verification (CCV) standard(s).

9.7.2 Instrument Tuning

At the beginning of every 12-hour shift when analyses are to be performed, the GC/MS system must be checked to see if acceptable performance criteria (Table VI) are achieved for DFTPP.

- 9.7.2.1 Inject 1 μ L of the 50 μ g/mL GC/MS tuning standard (see Section 7.2.1) into the GC/MS system.
- 9.7.2.2 The mass spectrum of the DFTPP must be obtained in the following manner: three scans (the peak apex scan and the scans immediately preceding and following the apex) are acquired and averaged. Background subtraction is also required, and must be accomplished using a single scan acquired no more than 20 scans prior to the elution of the DFTPP. Do not subtract part of the DFTPP peak. A procedure compliant with these requirements is programmed into a Macro used to evaluate the DFTPP spectrum. Confirm that all the key m/z criteria in Table VI are achieved.
- 9.7.2.3 If all the criteria are not achieved, the analyst must adjust or retune the mass spectrometer and repeat the test until all criteria are achieved. The performance criteria must be achieved before any samples, blanks, or standards are analyzed.

9.7.3 Initial Calibration (ICAL)

- **9.7.3.1** A new calibration curve must be generated initially, after major changes to the system, or when continuing calibration criteria cannot be met. Major changes include installation of new columns and source maintenance.
- 9.7.3.2 A minimum five-point initial calibration curve must be established for linear fit calibrations (weighted or unweighted). Six points or more are required for second order curve fits. See section 9.7.4 for Calibration Acceptance Criteria.

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The concentrations of standards commonly used to construct the PAH calibration curve are 20,100, 300, 600 (often analyzed before the rest of the standards and called the ICIS), 1200, 2500, and 5000 ng/mL.

- 9.7.3.3 If the concentration of any target compound in a sample exceeds the calibration range, the extract must be diluted so that the concentrations of all target compounds fall within the range of the calibration curve, and be reanalyzed. Any samples analyzed immediately following the sample that exceeded the linear range may require reanalysis due to possible carryover from the high-level sample.
- 9.7.3.4 Generally, it is NOT acceptable to remove points from a calibration for the purposes of meeting calibration criteria, unless the points are the highest or lowest on the curve AND the reporting limit and/or the linear range is adjusted accordingly. The only exception is that a level may be removed from the calibration if the reason can be clearly documented, for example a broken vial. A minimum of five levels must remain in the calibration. The documentation must be retained with the initial calibration. Alternatively, if the analyst believes that a point on the curve is inaccurate, the point may be reanalyzed and the reanalysis used for the calibration. All initial calibration points must be analyzed without any changes to instrument conditions, and all points must be analyzed within 12 hours.
- **9.7.3.5** Calculate the response factor (RF) for each analyte for each calibration standard level as described in Section 10.2. Calculate the mean RF and relative standard deviation (RSD) for each analyte as described in Section 11.3, respectively.

9.7.4 Calibration Acceptance Criteria and Corrective Action:

Acceptance Criteria 8270C:

The RSD of the initial calibration for each analyte of interest must be \leq 35%. (Refer to SOP DV-QA-024P for requirements for federal programs).

Acceptance Criteria 8270D:

Refer to Table VII for the acceptance criteria for minimum response factor and RSD. Two target compounds and surrogates may fail to meet the minimum RRF criteria listed in Table VII but must still meet the minimum RRF criteria of 0.010 (excluding compounds with a minimum RRF requirement of 0.010). In addition, two target compounds and surrogates may fail to meet the RSD criteria listed in Table VII but must still meet the maximum RSD requirement of 40%. (excluding compounds with a maximum RSD requirement of 40%). Refer to SOP DV-QA-024P for requirements for federal programs.

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Corrective Actions:

If these criteria cannot be met, least-squares weighted or unweighted linear regression may be used to establish a calibration function as described in Section 11.4. In this case, the correlation coefficient (r) must be greater than 0.995 ($r^2 > 0.990$) or a second-order regression fit with coefficient of determination (COD) greater than 0.990 may be used. If these linearity criteria are not achieved, verify the standard preparation and instrument conditions, and then recalibrate the instrument. If technical acceptance criteria are not met, it may be necessary to clean the ion source, perform injector maintenance, change the column, or take other corrective actions.

9.7.4.1 In the event that a least-squares regression is used, the analyst should evaluate the bias at the lower portion of the curve. This can be accomplished by re-fitting the low point standard back into the curve. The recalculated concentration should be within ±50% of the standard's true concentration. If these criteria are not met, the analyst may have to evaluate the concentration range of the standards, or the lower limit of quantitation.

9.8 Initial Calibration Verification (ICV)

The Initial Calibration Verification (ICV) is a second-source, mid-level standard that is analyzed immediately following the initial calibration standards.

Acceptance Criteria: The absolute value of the difference between the measured PAH

analyte concentration and the true value must be \leq 30 %.

Corrective Action: If the ICV recovery fails, then take the following actions:

Verify standard preparation, and if incorrect, re-prepare the ICV

standard solution.

If preparation of the ICV standard was correct, then re-prepare

the initial calibration standards and recalibrate.

Document all actions taken.

9.9 Continuing Calibration Verification (CCV)

Every 12 hours, the mass spectrometer response for each PAH relative to the internal standard is determined by analyzing a standard with the same analytes, concentrations, and lot numbers as the 600 ng/mL calibration standard. The RF for each compound in the continuing calibration verification (CCV) analysis is compared to the RF for that compound in the ICAL.

Acceptance Criteria 8270C:

The absolute value of the difference between the CCV RF for each PAH analyte and the corresponding ICAL value must be \leq 35 %. (Refer to SOP DV-QA-024P for requirements for federal programs).

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Acceptance Criteria 8270D:

The absolute value of the difference between the CCV RF for each PAH analyte and the corresponding ICAL value must meet the criteria in Table VII. The compounds must also meet the minimum response factor criteria listed in Table VII. Two target compounds and surrogates may fail to meet the minimum RRF criteria in Table VII (excluding compounds with a minimum RRF requirement of 0.010) but must still meet the minimum RRF criteria of 0.010. In addition, two target compounds and surrogates may fail to meet the difference criteria in Table VII (excluding compounds with a maximum percent difference requirement of $\pm 40\%$) but must still meet the maximum difference requirement of $\pm 40\%$. (Refer to SOP DV-QA-024P for requirements for federal programs).

Acceptance Criteria 8270C & 8270D:

The internal standard response of the CCV must be within 50-200% of the internal standard response in the corresponding level of the most recent ICAL sequence.

The internal standard retention time must be within \pm 30 seconds of the internal standard retention time in the corresponding level of the most recent ICAL sequence.

Corrective Action:

If, for any analyte, the CCV RF does not meet the stipulated acceptance criteria, a five-point calibration curve must be repeated for that analyte prior to the analysis of samples.

If any internal standard retention time in the CCV changes by more than 30 seconds from that of the corresponding level of the most recent ICAL sequence, the chromatographic system must be inspected for malfunctions and corrections made, as required.

10.0 Procedure

One-time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using an NCM. The NCM is automatically sent to the laboratory supervisor and Project Manager by e-mail so that the client can be notified as appropriate. The NCM process is described in more detail in SOP DV-QA-0031. The NCM shall be filed in the project file and addressed in the case narrative.

10.1 <u>Sample Preparation</u>

10.1.1 Aqueous Sample Extraction and Concentration

- **10.1.1.1** Instructions for the extraction of aqueous samples may be found in SOPs DV-OP-0006 or DV-OP-0008.
- **10.1.1.2** Instructions for the concentration of extracts may be found in SOP DV-OP-0007.

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10.1.2 Soil Sample Extraction and Concentration

- **10.1.2.1** Instructions for the ultrasonic extraction of soil samples may be found in SOP DV-OP-0016.
- **10.1.2.2** Instructions for the microwave extraction of soil samples may be found in SOP DV-OP-0015.
- **10.1.2.3** Instructions for the concentration of extracts may be found in SOP DV-OP-0007.

10.2 **Sample Analysis**

- 10.2.1 All aliquotting, extract dilutions, and spike additions must be performed in the trace fume hood using equipment dedicated to PAH-SIM analysis. An aliquot of each sample extract is placed into a two-milliliter GC/MS autosampler vial. Sufficient volume of extract remains should reanalysis be necessary.
- **10.2.2** Prior to analysis, internal standard is added to the sample vial giving a final internal standard concentration of 600 ng/mL in the extract.
- 10.2.3 Representative aliquots are injected into the gas chromatograph/mass spectrometer using similar conditions to those summarized in Table I. The injection volume is 1 μ L.
- **10.2.4** Whenever an unusually concentrated sample is encountered, it may be necessary to reanalyze the subsequent sample extracts after analyzing an instrument blank to demonstrate that there is no cross contamination.
- **10.2.5** The following is a typical analytical sequence:
 - Solvent rinses, as needed
 - MS tune
 - ICAL plus ICV or CCV
 - Instrument blank
 - MB. LCS
 - LCSD (if requested by client)
 - Sample extracts
 - MS and MSD are interspersed with sample extracts, and usually run after the sample from which they are produced.
 - The last sample extract must be run within 12 hours of the tune.
- 10.2.6 The sequence may be altered to accommodate reanalysis or additional instrument blank and calibration evaluations. At a minimum, an instrument blank or a method blank shall be included in the sequence. Refer to QC policy DV-QA-003P for additional details.
- **10.2.7** The effluent from the GC capillary column is fed directly into the ion source of the mass spectrometer. The MS is operated in the selected ion monitoring (SIM)

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mode using appropriate windows to include the quantitation and confirmation masses for each analyte as shown in Table IV.

- **10.2.8** All compounds detected at concentrations above the method MDL are checked to ensure that the confirmation ion is present at the appropriate ratio.
- 10.2.9 All compounds detected at concentrations above the highest calibration standard require dilution and reanalysis. In addition, any samples that were analyzed immediately following a high-level sample should be reanalyzed to rule out carryover from the high-level sample, unless they are preceded by an acceptable instrument blank or the high compound(s) were not detected in the subsequent samples.

10.2.10 Manual Integrations

Upon completion of the analytical sequence, transfer the raw instrument data to Target DB for further processing. Review the chromatograms to ensure correct assigning of peaks and correct integration of each peak.

Note that certain compounds (e.g., benzo(b)fluoranthene and benzo(k)fluoranthene) may require frequent manual integrations. Special attention must be exercised by the analyst and secondary reviewer for compounds that are commonly mis-integrated in automated software or are manually integrated. If manual data manipulations are necessary, they must be justified and documented. See DV-QA-011P requirements for manual integration.

11.0 Calculations / Data Reduction

11.1 Qualitative Identification

Obtain electronic ion current profiles (EICP) for the primary m/z and the confirmatory ion for detected compounds. The following criteria must be met to make a qualitative identification:

- **11.1.1** The characteristic masses of each parameter of interest must maximize in the same or within one scan of each other.
- **11.1.2** The retention time (RT) of unknown peaks must fall within \pm 0.2 minute of the RT for the compound in the daily calibration standard (mid-point ICAL or daily CCV).
- 11.1.3 The relative peak areas of the primary ion compared to the confirmation or secondary ion masses in the EICPs must fall within \pm 20% of the relative intensities of these masses in a reference mass spectrum. The reference mass spectrum can be obtained from a standard analyzed in the GC/MS system or from a reference library. A compound that does not meet secondary ion confirmation criteria may still be determined to be present in a sample after close inspection of the data by the mass spectroscopist. Supportive information includes correct relative retention time (RRT) and the presence of the secondary ion, but the ratio falls outside of \pm 20% of the primary ion, which may be caused by an interference of the secondary ion.

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11.1.4 Structural isomers that have very similar mass spectra and less than a 30second difference in retention time, can be explicitly identified only if the resolution between authentic isomers in a standard mix is acceptable. Acceptable resolution is achieved if there is a definitive inflection between the two peaks, according to the analyst's judgment. Otherwise, structural isomers are identified as isomeric pairs.

11.2 Detailed information regarding calibration models and calculations can be found in Corporate SOP CA-Q-S-005, Calibration Curves (General) and the public folder Arizona Calibration Training.

11.3 **Average Response Factor Calibration**

The following formula is used to calculate the response factor for each analyte of interest relative to the applicable internal standard for each of the calibration standards:

$$RF = \frac{A_s \times C_{is}}{A_{is} \times C_s}$$

Where:

As = Area of the characteristic ion for the target analyte in the calibration standard.
 A_{is} = Area of the characteristic ion for the internal standard.
 C_{is} = Concentration of the internal standard, (ng/mL).

Concentration of the target analyte in the calibration standard (ng/mL).

The calibration uses the average response factor for each target analyte, which is calculated as follows:

average (mean) RF =
$$\overline{RF} = \frac{\sum_{i=1}^{n} RF_i}{n}$$

Where:

 RF_i Response factor for the ith calibration level.

Number of calibration levels.

The standard deviation for the mean RF for each target analyte is calculated as follows:

$$SD = \sqrt{\frac{\sum_{i=1}^{n} \left(RF_i - \overline{RF} \right)^2}{n-1}}$$

The relative standard deviation (RSD) for the average response factor for each target analyte is calculated as follows:

$$RSD = \frac{SD}{RF} \times 100\%$$

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The concentration of each target analyte in the sample extract is calculated using the average response factor that was calculated in Section 0 as follows:

$$C_e = \frac{A_e \times C_{is}}{A_{is} \times \overline{RF}}$$

Where:

C_e = Concentration of target analyte in the sample extract, ng/mL.

A_e = Area of the characteristic ion for the target analyte in the sample extract.

= Area of the characteristic ion for the internal standard.

 C_{is} = Concentration of the internal standard, (ng/mL).

 \overline{RF} = Average response factor for the target analyte as determined by calibration.

11.4 Linear Least-Squares Regression Calibration (Unweighted)

A linear least-squares regression is performed using the concentration of the target analyte in the calibration standard as the independent variable (x) and the instrument response as the dependent variable (y). The regression produces the slope and intercept terms for a linear equation in the following form:

$$y = mx + b$$

Where:

y = instrument response (e.g., peak area)

x = concentration of target analyte in calibration standard

m = slope of the line b = intercept of the line

For the internal standard calibration, the regression equation is rewritten as follows:

$$\frac{A_s C_{is}}{A_{\cdot}} = mC_s + b$$

Where:

A_s = Area of the characteristic ion for the target analyte in the calibration standard.

 A_{is} = Area of the characteristic ion for the internal standard.

C_s = Concentration of the target analyte in the calibration standard, (ng/mL).

 C_{is} = Concentration of the internal standard, (ng/mL).

m = slope of the line

b = intercept of the line

The concentration in an unknown extract is then calculated by rearranging the calibration equation as follows:

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$$C_e = \frac{\left[\frac{A_s C_{is}}{A_{is}} - b\right]}{m}$$

Where C_e is the concentration of the target analyte in the sample extract, and A_e is the area of the characteristic ion for the target analyte in the sample extract.

The actual sample concentration (C) for each compound is calculated as follows:

$$C = C_e \times \left(\frac{V_e}{V_o}\right) \times DF$$

Where:

C = Concentration of the target analyte in the original sample,

ng/L (aqueous sample) or ng/kg (solid sample).

C_e = Concentration of the target analyte in the sample extract,

ng/mL.

V_e = Final extract volume, mL.

 V_o = The original volume or weight of the sample that was

extracted, L (aqueous sample) or kg (solid sample).

DF = Dilution factor, if appropriate.

11.5 Additional Regression Calibration Models

As needed, weighted linear least-squares or second order regressions may be utilized for this analysis. See Corporate SOP CA-Q-S-005, *Calibration Curves (General)*, Attachment 1, and the public folder *Arizona Calibration Training* for calculations and further explanations.

11.6 A second-level technical review of the organic data is performed prior to data reporting. This review is performed by a peer or supervisor using the guidelines and checklists detailed in SOP DV-QA-0020.

12.0 Method Performance

12.1 Method Detection Limit Study (MDL)

A valid method detection limit (MDL) must be determined for each analyte of interest prior to analyzing samples and must be verified at least annually thereafter. MDLs are determined using laboratory reagent water (for aqueous samples) and sand (for solid samples) that is spiked with the target analytes at concentrations near the estimated MDL. The MDL must be below the reporting limit for each analyte. The procedure for determination of the method detection limit is given in 40 CFR Part 136, Appendix B, and further defined in TestAmerica Denver Policy DV-QA-005P.

12.2 <u>Demonstration of Capabilities</u>

Each analyst must successfully complete an initial demonstration of capability (DOC) prior to analyzing samples. Demonstrations of capability for both soil and water matrices are

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required. On-going DOCs must be performed by each analyst annually. This requires analysis of QC check samples containing all of the standard analytes for the method. For some tests it may be necessary to use more than one QC check mix to cover all analytes of interest. Details for the initial and on-going DOCs are provided in SOP DV-QA-0024.

12.3 Training Requirements

The group/team leader has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use and has the required experience. Training and qualification requirements are detailed in SOP DV-QA-0024.

12.4 Retention Time Study

- **12.4.1** Expected absolute retention times (RTs) are initially determined by analyzing all target analytes in the open-scan mode. Example RTs are listed in Table V.
- **12.4.2** Relative retention times (RRTs) are then calculated for samples in each analytical run based on the RTs found in the continuing calibration verification standard (CCV).
- 12.4.3 RTs are re-established after any significant instrument maintenance, including source cleaning and changing columns, or whenever compounds are not adequately detected in CCVs or LCSs.

13.0 Pollution Control

Standards and reagents are prepared in volumes consistent with laboratory use to minimize the volume of expired standards and reagents requiring disposal.

14.0 Waste Management

- 14.1 All waste will be disposed of in accordance with Federal, State, and local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this procedure, the policies in section 13, "Waste Management and Pollution Prevention", of the Environmental Health and Safety Manual, and DV-HS-001P, "Waste Management Program."
- **14.2** The following waste streams are produced when this method is carried out:
 - **14.2.1** Expired Chemicals/Reagents/Standards Contact Waste Coordinator
 - **14.2.2** Methylene chloride solvent rinse waste Waste Stream B
 - **14.2.3** Expired extract vial waste Waste Stream A
 - 14.2.4 Radioactive and potentially radioactive waste must be segregated from non-radioactive and mixed waste as appropriate. Contact the Radioactive Waste Coordinator for proper management of radioactive or potentially radioactive waste generated by this procedure.

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15.0 References / Cross-References

- 15.1 Test Methods for Evaluating Soil Waste Physical/Chemical Methods (SW-846), Third Edition, September 1986, Final update I, July 1992, Final Update IIA, August 1993, Final Update II, September 1994; Final update IIB, January 1995; Final Update III, December 1996, Final Update IV January 2008.
 - **15.1.1** Method 8000B, Determinative Chromatographic Separations, Revision 2, December 1996.
 - **15.1.2** Method 8270C, Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS), Revision 3, December 1996.
 - **15.1.3** Method 8000C, Determinative Chromatographic Separations, Revision 2, February 2007.
 - **15.1.4** Method 8270D, Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS), Revision 4, February 2007.
 - **15.1.5** Method 3510C, Separatory funnel Liquid-Liquid Extraction, Revision 3, December 1996.
 - **15.1.6** Method 3520C, Continuous Liquid-Liquid Extraction, Revision 3, December 1996.
 - **15.1.7** Method 3550B. Ultrasonic Extraction. Revision 2. December 1996.
 - **15.1.8** Method 3546, Microwave Extraction, Revision 0, February 2006.
- **15.2** CLP Statement of work for Multi-Media, Multi-Concentration Organics Analysis, SOM01.2. June 2007.

16.0 <u>Method Modifications</u>

- 16.1 The CLP SOW referenced in 8270D does not require the analysis of DFTPP prior to the analysis of samples. The method relies on the successful analysis of calibration standards to verify acceptable function of the mass spectrometer. TestAmerica Denver utilizes the DFTPP check to identify any operational issues with the mass spectrometer prior to the analysis of the calibration standards. This allows the analyst to identify possible problems independent of the GC. As a result, the laboratory will start the 12 hour clock with the injection of the DFTPP, not the calibration standard as required in the method.
- Method 8270C serves as the basis for this SOP, but the method has been modified extensively for low-level analysis using selected ion monitoring (SIM) and optimizing instrument conditions for the low-level analysis. Consequently the sensitivity of the method has been enhanced and it is not uncommon to detect low-level contamination in the method blank at levels well below the limits of detection for the less sensitive GC/MS method. For example, Method 8270C states that the RSD of the initial and continuing calibration must less than or equal to 15% and 20% respectively. Due to the low-level nature of the analysis, this SIM procedure allows both of these criteria to be less than or equal to 35%.

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16.3 Method 8270C stipulates qualitative identification based on relative retention time (RRT), which is calculated by dividing the retention time (RT) of the target analyte by the RT of the internal standard. The RRT of the suspected target analyte in the sample extract must be within \pm 0.06 RRT units of the RRT for that analyte in the calibration standard. This SOP stipulates qualitative identification based on an absolute RT. Namely the RT of the suspected target analyte in the sample extract must be within \pm 0.2 minute of the RT for that analyte in the calibration standard. Additionally, the RT for the internal standard in the sample extract must also be within \pm 0.2 minute of the RT for the internal standard in the calibration standard. The criteria used in this SOP are more restrictive than those imposed by the referenced method. For the earliest eluting compounds, the RT for the internal standard is typically 8 minutes. The earliest eluting target analyte must be at a RRT of at least 0.8, which translates to a RT of 6.4 minutes. Assuming a worst-case scenario where the RT of the internal standard is 0.2 minute higher (i.e., 8.2 minutes) and the RT of the target analyte is 0.2 minute lower (i.e., 6.2 minutes), the calculated RRT is 0.76. The total deviation from the expected RRT is 0.04 RRT units, which is smaller than what is allowed by Method 8270C.

17.0 Attachments

Table I: Routine Instrument Operating Conditions
Table II: Surrogates for Standard List Analysis

Table III: Internal Standards for Standard List Analysis Table IV: PAH Compounds and Ions Used for Analysis

Table V: Example Retention Times, IS and Surrogate Associations

Table VI: DFTPP Key Ions and Ion Abundance Criteria for 8270C and 8270D Table VII: 8270D Relative Response Factor Criteria for Initial and Continuing

Calibration

Appendix I: Extended List PAHs

18.0 Revision History

- Revision 7: 31 July 2012
 - Annual Technical Review
 - Grammatical and formatting changes throughout
 - Updated the quant ion for surrogate terphenyl-d14 to IS#2 in Table V
 - Updated Table 1 to match current GC conditions
- Revision 6.2: 31 August 2011
 - Inserted Section 7.2.4.
 - Revised QC section (Section 9)
 - o Inserted paragraph 10.2.10. regarding manual integration
 - o Added Section 11.5
 - Revised Section 16.2 regarding calibration criteria
 - Updated prep methods used and inserted prep methods in reference section
 - o Annual Technical Review
 - o Grammatical and formatting changes throughout
- Revision 6.1: August 2010.
 - Annual Technical Review

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- Revision 6: August 2009.
 - o Removed the requirement to have PCP in the DFTPP tune standard.
 - Calibration sections were updated to reflect the criteria in method 8270D and the CLP Multi-media and Multi-concentration for Organic Analysis.
 - o Section 2 on instrument conditions was expanded.
 - o Table VII was added.
- Revision 5.1: June 2009.
 - o Added Appendix I for extended list PAHs.
 - o Removed all references to Pentachlorophenol (PCP), the lab no longer supports this method for the analysis of PCP.
- Revision 5: April 2008.
 - Revised calibration levels in section 7.4 to include the low level standard for the initial calibration.
 - Changed references to TestAmerica and adjusted format to comply with TestAmerica format.

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Table I: Routine Instrument Operating Conditions

GC Conditions	
Inlet	Pulsed Split at 275 °C
Capillary Column	Varian Vf-5MS, 30 m length, 0.25 mm diam ID, 0.5 μm thickness
Column Mode	Constant flow, 3.4 mL/min
Temperature Program	Initial temp = 50 °C 25 °C/min ramp to 170 °C
	35 °C/min ramp to 325 °C and hold for at least 1 minute past the elution time of the last compound.
Run Time	About 20 minutes with a new column.
Carrier Gas	Helium Purge flow = 25.0 mL/min, 3.00 min Total flow ≈ 31 mL/min
Injection Volume	1.0 μL
Transfer Line	290 °C or 300 °C
Mass Spectrometer Conditions	
MS Source	230 °C or 240 °C
MS Quadrupole	200 °C
Dwell Time per Ion	Ranges from 30 to 100 milliseconds
lons	See following tables

NOTE:

The conditions listed above are subject to final fine adjustments to maximize instrument sensitivity. Changes to the above conditions are acceptable as long as method criteria are met.

Table II: Surrogates for Standard List Analysis

PAH Surrogates	Mass Ion	Confirmation Ion
Nitrobenzene-d ₅	82	128
2-Fluorobiphenyl	172	171
Terphenyl-d ₁₄	244	122

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Table III: Internal Standards for Standard List Analysis

Compound	Mass Ion	Confirmation Ion
Acenaphthene-d ₁₀	164	162
Phenanthrene-d ₁₀	188	94
Chrysene-d ₁₂	240	120

Table IV: PAH Compounds and Ions Used for Analysis

Compound	Mass Ion	Confirmation Ion
Acenaphthene	153	152
Acenaphthylene	152	151
Anthracene	178	179
Benzo(a)anthracene	228	229
Benzo(a)pyrene	252	253
Benzo(b)fluoranthene	252	253
Benzo(g,h,i)perylene	276	277
Benzo(k)fluoranthene	252	253
Chrysene	228	226
Dibenzo(a,h)anthracene	278	139
Dibenzofuran	168	139
Fluoranthene	202	101
Fluorene	166	165
Indeno(1,2,3,cd)pyrene	276	138
1-Methylnaphthalene	142	115
2-Methylnaphthalene	142	115
Naphthalene	128	129
Phenanthrene	178	179
Pyrene	202	101

Table V: Example Retention Times, IS and Surrogate Associations

Compound	RT (min.)	IS#	Surrogate #
Naphthalene	5.921	1	1
2-Methylnaphthalene	6.595	1	1
1-Methylnaphthalene	6.700	1	1
Acenaphthylene	7.512	1	2
Acenaphthene	7.686	1	2
Dibenzofuran	7.861	1	2
Fluorene	8.210	1	2
Phenanthrene	9.194	2	2
Anthracene	9.255	2	2
Fluoranthene	10.768	2	2
Pyrene	11.166	2	2
Benzo(a)anthracene	13.827	3	3
Chrysene	13.924	3	3
Benzo(b)fluoranthene	17.004	3	3
Benzo(k)fluoranthene	17.089	3	3
Benzo(a)pyrene	18.034	3	3
Indeno(1,2,3,cd)pyrene	21.509	3	3
Dibenz(a,h)anthracene	21.583	3	3
Benzo(g,h,i)perylene	22.306	3	3
Acenaphthene-d ₁₀	7.657	1	-
Phenanthrene-d ₁₀	9.177	2	-
Chrysene-d ₁₂	13.856	3	-
Nitrobenzene-d ₅	5.201	1	1
2-Fluorobiphenyl	6.945	1	2
Terphenyl-d ₁₄	11.38	2	3

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Table VI: DFTPP Key Ions and Ion Abundance Criteria 8270C

Mass	Ion Abundance Criteria
51	30-60 % of mass 198
68	< 2 % of mass 69
69	Mass 69 relative abundance
70	< 2 % of mass 69
127	40-60 % of mass 198
197	< 1 % of mass 198
198	Base peak, 100 % relative abundance
199	5-9 % of mass ion 198
275	10-30 % of mass 198
365	> 1 % of mass 198
441	Present, but less than mass 443
442	40-100 % of mass 198
443	17-23 % of mass 442

With the exception of mass 442, the tune criteria for SW846 method 8270D are less stringent for the criteria required in SW846 method 8270C. For 8270D, the 442 mass must be greater than 50% of mass 198 to meet the tune criteria. By using the 8270C criteria, the rest of the data will be within the 8270D criteria.

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Table VII: 8270D Relative Response Factor Criteria for Initial and Continuing Calibration

Compound	Minimum RRF	Maximum %RSD	Maximum %Diff
Acenaphthene	0.900	20	25
Acenaphthylene	0.900	20	25
Anthracene	0.700	20	25
Benzo(a)anthracene	0.800	20	25
Benzo(a)pyrene	0.700	20	25
Benzo(b)fluoranthene	0.700	20	25
Benzo(g,h,i)perylene	0.500	20	25
Benzo(k)fluoranthene	0.700	20	25
Chrysene	0.700	20	25
Dibenzo(a,h)anthracene	0.400	20	25
Dibenzofuran	0.800	20	25
Fluoranthene	0.600	20	25
Fluorene	0.900	20	25
Indeno(1,2,3,cd)pyrene	0.500	20	25
1-Methylnaphthalene	0.400	20	25
2-Methylnaphthalene	0.400	20	25
Naphthalene	0.700	20	25
Phenanthrene	0.700	20	25
Pyrene	0.600	20	25

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Appendix I: Extended List PAH Analysis by GC/MS

Summary of Method

This is the extended list for the SIM analysis that some clients require.

Modifications from the SIM analysis are as follows:

- The DFTPP tune has tailing factors that are calculated for Pentachlorophenol and Benzidine and a DDT breakdown check is performed.
- The instrument is calibrated at eight concentration levels. The calibration levels are made by diluting two stock standards with concentrations of 20 μg/mL [PAHXSIM stock (#1)] and 2μg/mL [PAHXSIM 2° stock (#2)] down to the concentrations listed below, in methylene chloride. All phthalate compounds and 2-methylnaphthalane are at a ratio of 2:1 in the stock standards. Therefore, if the concentration is 0.02μg/mL for the target analytes, the phthalates are at 0.04μg/mL.

Level (μg/mL)	Stock ID	Stock Amt (μL)	Solvent amount (μL)	IS amount (μL)	Final Volume (μL)
0.02 μg/mL	#2	5	495	50	500
0.1 μg/mL	#2	25	475	50	500
0.3 μg/mL	#2	75	425	50	500
0.6 μg/mL	#1	15	485	50	500
1.2 μg/mL	#1	30	470	50	500
2.5 μg/mL	#1	62.5	437.5	50	500
5.0 μg/mL	#1	125	375	50	500
10.0 μg/mL	#1	250	250	50	500

Response factors for each compound must be ≤20% RSD. If any compound is >20% RSD, must use the best curve fit.

Initial Calibration Verification

The second source calibration stock is also at 20 μg/mL (PAHSIM SSV stock).

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- The second source verification (SSV or ICV) is analyzed at 1.2 μg/mL.
- The acceptance criterion for the ICV is ±25%D.

Continuing Calibration Verification

- The CCV is run at 0.6μg/mL
- The criterion: The Average %D for all compounds must be <20%D, with no single compound exceeding 30%D.

Sample extraction: See DV-OP-0008 (aqueous) and DV-OP-0009 (soil).

Sample concentration: See DV-OP-0007.

Internal Standard final concentration is 6 $\mu g/mL$ in standards and extracts. The stock is at 400 $\mu g/mL$

Control limits are stored in the LIMS system.

Target Analytes
Acenaphthene
Acenaphthylene
Anthracene
Benzo(a)anthracene
Benzo(k)fluoranthene
Benzo(b)fluoranthene
Benzo(g,h,i,)perylene
Benzo(a)pyrene
Dibenzo(a,h)anthracene
Fluoranthene
Indeno(1,2,3-cd)pyrene
2-Methylnaphthalene
1-Methylnaphthalene
Phenanthrene
Pyrene
Naphthalene
Fluorene
Chrysene
Hexachlorobutadiene
Hexachlorobenzene
Dibenzofuran

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Extended List Compounds
1,4 Dioxane
N-Nitrosodiphenylamine
N-Nitrosodimethylamine
Butyl Benzyl Phthalate
Dimethyl Phthalate
Diethyl Phthalate
Bis(2-Ethylhexyl) Phthalate
Di-n-octyl Phthalate
Di-n-butyl Phthalate

The recovery for the spike pair must be within the control limits stored in the LIMS. The MS/MSD pair is aliquotted and run two times on the instrument, to confirm the results. If the results to be reported are from the first analysis, it is not required that the second analysis be within the 12 hour tune clock.

Some of the above compounds have advisory limits (30-150% Recovery). There is not enough data to sufficiently determine accurate control limits. As sufficient data are collected (ideally at least 30 data points), the spike control limits will be updated to \pm 3 standard deviations of the historical mean % recovery. Limits will be updated annually.

The GCMS instrumentation is configured the same as in the SIM analysis, but the ramp parameters are the following:

Ramp	Rate (°C/min)	Temp Hold (°C)	Time of Hold (min)
		40	2
1	25	190	0
2	35	325	varies *

* The length of the last hold is dependent on the length of the column. This hold is typically 1-1 ½ minutes after the last compound elutes.



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Title: Determination of Volatile Organics by GC/MS [8260B and 624]

Approvals (Signature/Date):			
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1.0 Scope and Application

1.1 This method is applicable to the determination of volatile organic compounds (VOCs) in water, wastewater, soils, sludges, and other solid matrices. Standard analytes are listed in Table 1. Additional analytes that can be determined by this SOP are listed in Tables 2 and 3.

- 1.2 This SOP is applicable to Method 8260B, which is appropriate for compliance testing under RCRA regulations and Method 624 (CWA compliance testing). It is important that the procedural differences described in this document for these methods are carefully observed.
- **1.3** Appendix A of this SOP contains the modifications needed to run the instrument in the selected ion monitoring mode.
- 1.4 This method can be used to quantify most volatile organic compounds that have boiling points below 200 °C and are insoluble or slightly soluble in water. Volatile water-soluble compounds can be included in this analytical technique; however, for more soluble compounds, quantitation limits are approximately ten times higher because of poor purging efficiency.
- 1.5 The method is based upon a purge-and-trap, gas chromatograph/mass spectrometric (GC/MS) procedure. The approximate working range is 0.5 to 60 μ g/L for 8260B waters, 2.5 to 200 μ g/kg for low-level soils, and 200 to 30,000 μ g/kg for medium-level soils. The working range for Method 624 (5 mL purge) is 5-200 μ g/L.
- 1.6 Reporting limits for Method 8260B are listed in Tables 1, 2, and 3. Reporting limits for Method 624 and 8260B SIM are given in Table A1 and Table Ap-1, respectively. Reporting limits for soil samples prepared by the AK methanol technique are listed in Table Bp-1.
- **1.7** Method performance is monitored through the use of surrogate compounds, matrix spike/matrix spike duplicates (MS/MSD), and laboratory control spike samples (LCS).

2.0 **Summary of Method**

- 2.1 Volatile compounds are introduced into the gas chromatograph by the purge and trap method. The components are separated via the gas chromatograph and detected using a mass spectrometer, which is used to provide both qualitative and quantitative information.
- 2.2 Aqueous samples are purged directly. Generally, soils are preserved by extracting the volatile analytes into methanol. If especially low detection limits are required, soil samples may be preserved in water (with or without sodium bisulfate) and purged directly.
- 2.3 In the purge-and-trap process, an inert gas is bubbled through the solution at ambient temperature or at 40 °C (40 °C is required for low-level soils), and the volatile components are efficiently transferred from the aqueous phase to the vapor phase. The vapor is swept through a sorbent column where the volatile components are trapped. After purging is completed, the sorbent column (trap) is heated and backflushed with inert gas to desorb

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the components onto a gas chromatographic column. The gas chromatographic column is then heated to elute the components, which are detected with a mass spectrometer.

Qualitative identifications are confirmed by analyzing standards under the same conditions used for samples and comparing the resultant mass spectra and GC retention times. Each identified component is quantified by relating the MS response for an appropriate selected ion produced by that compound to the MS response for another ion produced by an internal standard.

3.0 <u>Definitions</u>

3.1 Terms

The quality control terms used in this procedure are consistent with SW-846 terminology. Definitions are provided in the glossary of the TestAmerica Denver Quality Assurance Manual (QAM) and in SOP DV-QA-003P, Quality Assurance Program.

3.2 Calibration Check Compound (CCC)

CCCs are a representative group of compounds that are used to evaluate initial calibrations and continuing calibrations. Relative percent difference for the initial calibration and percent drift for the continuing calibration response factors are calculated and compared to the specified method criteria.

3.3 System Performance Check Compounds (SPCC)

SPCCs are compounds that are sensitive to system performance problems and are used to evaluate system performance and sensitivity. A response factor from the continuing calibration is calculated for the SPCC compounds and compared to the specified method criteria.

3.4 Initial Calibration Verification (ICV)

The ICV is a second-source calibration verification standard. In this SOP, the LCS and the MS/MSD spikes are second-source standards.

3.5 Continuing Calibration Verification (CCV)

A solution of method analytes, surrogate compounds, and internal standards used to evaluate the performance of the instrument system with respect to a defined set of method criteria.

3.6 Selected Ion Monitoring (SIM)

Operation of the mass spectrometer in the selected ion monitoring mode to optimize the quantitative information at the expense of qualitative information gained from other methods of analysis.

4.0 Interferences

4.1 Method interferences may be caused by contaminants in solvents, reagents, glassware, and other processing apparatus that lead to discrete artifacts. All of these materials must

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be routinely demonstrated to be free from interferences under conditions of the analysis by running laboratory method blanks as described in the Quality Control section. The use of ultra high purity gases, pre-purged purified reagent water, and approved lots of purge-and-trap-grade methanol will greatly reduce introduction of contaminants. In extreme cases, the purging vessels may be pre-purged to isolate the instrument from laboratory air contaminated by solvents used in other parts of the laboratory.

- 4.2 Samples can be contaminated by diffusion of volatile organics (particularly methylene chloride and fluorocarbons) into the sample through the septum seal during shipment and storage. A field blank prepared from reagent water and carried through the sampling and handling protocol can serve as a check on such contamination.
- 4.3 Matrix interferences may be caused by non-target contaminants that are co-extracted from the sample. The extent of matrix interferences will vary considerably from source to source depending upon the nature and diversity of the site being sampled.
- 4.4 Cross-contamination can occur whenever high-level and low-level samples are analyzed sequentially or in the same purge position on an autosampler. Whenever an unusually concentrated sample is analyzed, it should be followed by one or more blanks to check for cross-contamination. The purge and trap system may require extensive bake-out and cleaning after a high-level sample.
- 4.5 Some samples may foam when purged due to surfactants present in the sample. When this kind of sample is encountered, an antifoaming agent (e.g., J.T. Baker's Antifoam B silicone emulsion) can be used. A blank spiked with this agent must be analyzed with the sample. (See Section 10.7.4.12.)
- 4.6 Interferences are observed with the surrogate Toluene-d₈ when the samples appear to be treated with potassium permanganate.

5.0 Safety

- **5.1** Employees must abide by the policies and procedures in the Environmental Health and Safety Manual, Radiation Safety Manual and this document.
- 5.2 This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, nitrile gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.3 Specific Safety Concerns or Requirements

- 5.3.1 The gas chromatograph and mass spectrometer contain zones that have elevated temperatures. The analyst needs to be aware of the locations of those zones, and must cool them to room temperature prior to working on them.
- 5.3.2 The mass spectrometer is under deep vacuum. The mass spectrometer must be brought to atmospheric pressure prior to working on the source.

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5.3.3 There are areas of high voltage in both the gas chromatograph and the mass spectrometer. Depending on the type of work involved, either turn the power to the instrument off, or disconnect it from its source of power.

5.4 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table. A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Methanol	Flammable Poison Irritant	200 ppm (TWA)	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness, and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.

- (1) Always add acid to water to prevent violent reactions.
- (2) Exposure limit refers to the OSHA regulatory exposure limit.

6.0 **Equipment and Supplies**

6.1 <u>Instrumentation</u>

- **6.1.1** Purge and Trap Device: The purge and trap device consists of the sample purger, the trap, and the desorber.
- 6.1.2 Sample Purger: The recommended purging chamber is designed to accept between 5 mL and 25 mL samples with a water column at least 3 cm deep. The purge gas must pass through the water column as finely divided bubbles, each with a diameter of less than 3 mm at the origin. The purge gas must be introduced no more than 5 mm from the base of the water column. Alternative sample purge devices may be used provided equivalent performance is demonstrated. Low level soils are purged directly from a VOA vial.
- Trap: A variety of traps may be used, depending on the target analytes required. The O.I. #10 (Tenax / Silica gel / Carbon Molecular Sieve) is recommended. Other traps such as the Vocarb 3000 or Vocarb 4000 may be used if the Quality Control criteria are met.
- 6.1.4 Desorber: The desorber should be capable of rapidly heating the trap up to 270 °C depending on the trap packing material. Many such devices are commercially available.

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- **6.1.5** Sample Heater: A heater capable of maintaining the purge device at 40 °C is necessary for low level soil analysis.
- **6.1.6** Purge-and-trap Autosampler: An autosampler capable of sampling from a sealed vial. Varian Archon, or equivalent.
- **6.1.7** Gas Chromatograph: The gas chromatograph (GC) system must be capable of temperature programming.
- **6.1.8** Gas Chromatographic Columns: Capillary columns are used. Some typical columns are listed below:
 - **6.1.8.1** Column 1: 60 m X 0.25 ID DB-624 with 1.4 µm film thickness.
 - 6.1.8.2 Column 2: 75 m X 0.53 ID DB-624 wide bore with 3 µm film thickness.
- 6.1.9 Mass Spectrometer: The mass spectrometer must be capable of scanning 35-300 amu every two seconds or less, using 70 volts electron energy in the electron impact mode and capable of producing a mass spectrum that meets the required criteria when 50 ng of 4-bromofluorobenzene (BFB) are injected onto the gas chromatograph column inlet.
- **6.1.10** GC/MS interface: In general, glass jet separators are used but any interface (including direct introduction to the mass spectrometer) that achieves all acceptance criteria may be used.
- 6.1.11 Data System: A computer system that allows the continuous acquisition and storage on machine-readable media of all mass spectra obtained throughout the duration of the chromatographic program. The computer must have software that allows searching any GC/MS data file for ions of a specified mass and plotting such ion abundances versus time or scan number. This type of plot is defined as an Extracted Ion Current Profile (EICP). Software must also be available that allows integrating the abundances in any EICP between the specified time or scannumber limits. In addition, for the non-target compounds, software must be available that allows for the comparison of sample spectra against reference library spectra. The most recent release of the NIST/EPA mass spectral library should be used as the reference library. The computer system must also be capable of backing up data for long-term off-line storage.

6.2 Computer Software and Hardware

6.2.1 Please refer to the master list of documents, software and hardware located on G:\QA\Read\Master List of Documents\Master List of Documents, Software and Hardware.xls (or current revision) for the current software and hardware to be used for data processing.

6.3 Supplies

- **6.3.1** Microsyringes: 10 μL and larger, 0.006-inch ID needle.
- **6.3.2** Syringe: 5 or 25 mL glass with Luerlok tip, if applicable to the purging device.

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- **6.3.3** Balance: Analytical balance capable of accurately weighing 0.0001 g, and a top-loading balance capable of weighing 0.1 g
- **6.3.4** Vials: 2 mL, 20 mL, and 40 mL with screw caps and Teflon liners
- **6.3.5** Disposable magnetic stirrers for low-level soil analyses
- **6.3.6** Volumetric flasks: 10 mL and 100 mL, class A with ground-glass stoppers.
- **6.3.7** Spatula: Stainless steel.
- **6.3.8** Disposable pipettes: Pasteur.
- **6.3.9** pH paper: Wide range.
- **6.3.10** Gases:
 - **6.3.10.1** Helium: Ultra high purity, grade 5, 99.999%.
 - **6.3.10.2** Compressed nitrogen: Used for instrument pneumatics.

7.0 Reagents and Standards

- **7.1** Methanol: Purge and Trap Grade, High Purity
- **7.2** Reagent Water: High purity water that meets the requirements for a method blank when analyzed. (See Section 9.3.) Reagent water may be purchased as commercial distilled water and prepared by purging with an inert gas overnight. Other methods of preparing reagent water are acceptable.
- **7.3** Sand: Reagent grade Ottawa sand or equivalent.
- **7.4** Antifoam B, Silicon Emulsion, J. T. Baker, 100% purity.
- **7.5** Sodium bisulfate (NaHSO₄), reagent grade
- **7.6** If stock or secondary dilution standards are purchased in sealed ampoules they may be used up to the manufacturers' expiration date.

7.7 Calibration Stock Standard Solutions

Stock solutions may be purchased as certified solutions from commercial sources or prepared from pure standard materials as appropriate. These standards are prepared in methanol and stored in Teflon-sealed screw-cap bottles with minimal headspace at -10 to -20 °C. Stock standards and aliquots for gases must be replaced at least every week. The Gas Standards Tracking Log is used to verify track open dates to assist in weekly replacement of the gas standards. See Attachment 1. Other stock standards must be replaced at least every 6 months.

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7.8 Calibration Working standards

A working solution containing the compounds of interest prepared from the stock solution(s) in methanol. These standards are stored in the freezer or as recommended by the manufacturer. Working standards are monitored by comparison to the initial calibration curve. If any of the calibration check compounds drift in response from the initial calibration by more than 20%, then corrective action is necessary. This may include steps such as instrument maintenance, preparing a new calibration verification standard or tuning the instrument. If the corrective actions do not correct the problem then a new initial calibration must be performed.

- **7.9** Aqueous calibration standards are prepared in reagent water using the secondary dilution standards. These aqueous standards must be prepared daily.
- **7.10** Internal standards (IS) are added to all samples, standards, and blank analyses. Refer to Tables 7 and 7A for internal standard components.
- **7.11** Surrogate Standards: Refer to Tables 8 and 8A for surrogate standard components and spiking levels.
- **7.12** Laboratory Control Sample Spiking Solutions: Refer to Table 9 for LCS components and spiking levels.
- **7.13** Matrix Spiking Solutions: The matrix spike contains the same components as the LCS. Refer to Table 9.
- 7.14 Tuning Standard: A standard is made up that will deliver 50 ng on column upon injection. A recommended concentration of 50 ng/µL of BFB in methanol is prepared from stock standards as described in Sections 7.7 and 7.8.

8.0 Sample Collection, Preservation, Shipment and Storage

8.1 Water samples

- 8.1.1 Water samples are collected in triplicate in 40 mL glass VOA vials with PTFE-lined septum caps with minimal headspace. There should be no bubbles present in the container larger than ~6 mm.
- 8.1.2 Preservation depends upon the target analytes and the sampling location. At a minimum, aqueous samples are stored refrigerated at ≤ 6 °C and not frozen. Specific preservation requirements are given in the following table. If multiple analytes are requested, it may be necessary to provide aliquots with different preservations. For each preservation technique, the samples should be collected in triplicate.
- 8.1.3 The State of Colorado Attorney General's office issued a letter on July 1, 1998 requiring that all samples collected for analysis of volatile organic compounds in groundwater must be collected without acid preservation. The letter explains that this is done to avoid effervescence with alkaline samples and loss of volatiles. The letter also explains that the holding time for unpreserved ground waters is 14 days.

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8.1.4 SW-846 states that if carbonaceous materials are present, or if MTBE and other fuel oxygenate ethers are present and a high temperature sample preparative method is to be used, do not acid preserve the samples. The holding time for these unpreserved samples is 7 days. SW-846 does not otherwise provide guidance for processing unpreserved samples. EPA MICE has interpreted the holding time on an unpreserved sample as 7 days.

Preservation and Holding Time for Volatiles in Water

Analyte(s)	Reference	Preservation ¹	Holding time	Dechlorination Required ²
Routine target analytes ³	SW-846, Ch. 4	Cool, <6°C, pH < 2 with 1:1 HCl	14 days	Y
	SW-846, Ch. 4	Cool, <6°C	7 days	Y
	624	Cool, <u><</u> 6°C, pH < 2 with 1:1 HCl	14 days	Y
	624	Cool, <u><</u> 6°C	7 days	Y
Acrolein ⁴	SW-846, Ch. 4	Cool, <u><</u> 6°C, pH 4-5	7 days	N
	603	Cool, <u><</u> 6°C (no HCl)	3 days	Υ
	603	Cool, <u><</u> 6°C, pH 4-5	14 days	Y
Acrylonitrile ⁴	SW-846, Ch. 4	Cool, <u><</u> 6°C, pH 4-5	7 days	N
	603	Cool, <u><</u> 6°C (no HCl)	14 days	Y
	603	Cool, <u><</u> 6°C, pH 4-5	14 days	Y
2-Chloroethylvinyl ether (2-CLEVE) ⁵	SW-846, Ch. 4	Cool, <6°C (no HCl)	7 days	Y
	624	Cool, <u><</u> 6°C (no HCl)	14 days	Y

¹ See Section 8.1.3 for samples collected in Colorado and Section 8.1.4 for samples to be analyzed by Method 8260B that are unpreserved.

² If residual chlorine is present, 2 drops of 10% sodium thiosulfate are added

Separate aliquots must be collected and preserved as indicated if acrolein, acrylonitrile, 2-CLEVE (by Methods 8260B or 624), vinyl chloride (by Method 8260B) or styrene (by Method 8260B) are also to be analyzed. If aromatic and biologically active compounds are analytes of interest, acid preservation is necessary.

⁴ According to the source methods, the preferred method for acrolein and acrylonitrile is Method 603. In the Method Update Rule published in the Federal Register on May 18, 2012 (40 CFR Parts 136, 260, et. al.) EPA approved Method 624 for the determination of acrolein and acrylonitrile in wastewater. The current sample preservation and holding time requirements for acrolein and acrylonitrile apply to these compounds when analyzed by Method 624. Implementation of this rule is subject to individual state program decisions and timetables.

⁵ SW-846 includes vinyl chloride and styrene in the list of compounds which require unpreserved sample for analysis. Method 624 does not include these two analytes on the standard analyte list.

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8.2 Soil Samples

8.2.1 Soil samples can be taken using the EnCore™ sampler. Typically three Encores are collected per sampling location. At specific client request, unpreserved soil samples may be accepted for preservation at the lab.

- 8.2.1.1 Samples sent in the EnCore[™] sampler to the lab for preservation must be preserved within 48 hours of sampling. They are preserved by extruding one sample into a clean VOA vial containing methanol for medium level analysis. The remaining two samples are extruded into vials containing water or sodium bisulfate (NaHSO₄) and water for low level analysis.
- **8.2.1.2** Samples are stored frozen after transfer from the EnCoreTM sampler.
- 8.2.2 The more common way to collect soils is with Terra Core kits. Typically three aliquots are collected. Terra Core kits consist of the Terra Core sampling device and three 40 mL tared VOA vials. There are several ways to preserve the samples once sampled.
 - **8.2.2.1** The samples collected with the Terra Core sampling device are extruded into empty vials and frozen in the field. The lab freezes the samples on receipt. These samples have a 14 day holding time from sampling.
 - 8.2.2.2 The samples can be extruded into empty vials and shipped to the lab refrigerated. The lab freezes the samples within 48 hours of collection and the holding time is extended to 14 days from collection. The lab has the option to prepare the samples upon receipt by the addition of methanol to one vial and water or sodium bisulfate (NaHSO₄) and water to the remaining two vials. The samples are then refrigerated. The holding time for this latter preservation is 14 days from sampling.
 - 8.2.2.3 Alternatively, the project team can request for each sample one tared vial containing methanol for medium level analysis and two tared vials containing water or sodium bisulfate and water, depending upon project requirements. An aliquot of the sample is extruded into each prepared vial while in the field and shipped on ice. The samples are refrigerated upon receipt at the laboratory. The holding time is 14 days from sampling for this field preservation technique.

8.2.3 Unpreserved Soils

8.2.3.1 At specific client request unpreserved soils packed into glass jars or brass tubes may be accepted and subsampled in the laboratory. This is the old procedure based on Method 5030A. It is no longer included in subsequent revisions of Method 5030 and is likely to generate results that are biased low, possibly by more than an order of magnitude.

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- **8.2.3.2** The maximum holding time is 14 days from sampling until the sample is analyzed. Unpreserved samples should be analyzed as soon as possible. The lack of preservation should be addressed in the case narrative.
- **8.2.4** An additional bottle of unpreserved soil for each sampling location must be shipped for percent moisture determination.
- **8.2.5** A second bottle of unpreserved soil is sent for screening.
- **8.2.6** Preservation and holding times for volatiles in soils are summarized in the following table, based on SW-846 Method 5035A. The "Coring Tool" listed in the container column may be the EnCoreTM or Terra Core sampler.

Preservation and Holding Time for Volatiles in Soil Method 5035A

Container/Contents ¹	Preservation	Holding time	Analysis
Empty Sealed Vial	Freeze on-site to -7°C (do not freeze below -20°C)	14 days	Low Level
Empty Sealed Vial	Cool to ≤ 6 °C	48 hours	Low Level
Empty Sealed Vial	Cool to ≤ 6 °C for no more than 48 hours Frozen upon receipt at lab (< -7 °C, do not freeze below -20 °C)	14 days	Low Level
Empty Sealed Vial	Cool to ≤ 6 °C for no more than 48 hours Preserved with methanol upon receipt at lab	14 days	Medium Level
Encore TM sampler used for transport	Cool to ≤ 6 °C or Freeze to < -7 °C in field	48 hours	Low or medium level
Encore [™] sampler used for transport	Cool to \leq 6°C or freeze to $<$ -7°C in field and upon receipt at lab extruded to a sealed vial and either frozen to $<$ -7°C or chemically preserved	14 days	Low or medium level
Vial containing reagent water	Sample is extruded into vial and frozen to < -7°C in field and maintained frozen upon receipt by the laboratory.	14 days	Low level
Vial containing reagent water	Sample is extruded into vial and cooled to \leq 6°C in field then frozen to $<$ -7°C upon laboratory receipt (within 48 hours of sampling).	14 days	Low level
Vial containing reagent water and 1 g NaHSO ₄ ²	Sample is extruded into vial with preservative and cooled to \leq 6°C. Stored at \leq 6°C upon laboratory receipt.	14 days	Low Level
Vial containing methanol	Sample is extruded into vial with preservative, cooled to $\leq 6^{\circ}$ C and frozen upon receipt at laboratory.	14 days	Medium Level

¹ For biologically active soils, immediate chemical or freezing preservation is necessary due to the rapid loss of BTEX compounds within the first 48 hours of sample collection.

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Reactive compounds such as 2-chloroethylvinyl ether readily break down under acidic conditions. If these types of compounds are analytes of interest, collect a second set of samples without acid preservatives and analyze as soon as possible

- **8.3** Trip blanks, consisting of laboratory prepared water samples with acid preservative, are also provided when bottles are supplied by the laboratory to the field. Trip blanks are used for both water and soil samples to monitor potential contamination from volatile compounds in transit and in the field.
- A holding blank is stored in each refrigerator with the samples. This is analyzed every 7 14 days (see SOP DV-QA-0013).

9.0 **Quality Control**

- **9.1** The minimum quality controls (QC), acceptance criteria, and corrective actions are described in this section. When processing samples in the laboratory, use the LIMS Method Comments to determine specific QC requirements that apply.
 - **9.1.1** The laboratory's standard QC requirements, the process of establishing control limits, and the use of control charts are described more completely in TestAmerica Denver policy DV-QA-003P, Quality Assurance Program.
 - 9.1.2 Specific QC requirements for Federal programs, e.g., Department of Defense (DoD), Department of Energy (DOE), AFCEE, etc., are described in TestAmerica Denver policy DV-QA-024P, Requirements for Federal Programs.
 - 9.1.3 Project-specific requirements can override the requirements presented in this section when there is a written agreement between the laboratory and the client, and the source of those requirements should be described in the project documents. Project-specific requirements are communicated to the analyst via Method Comments in the LIMS and the Quality Assurance Summaries (QAS) in the public folders.
 - 9.1.4 Any QC result that fails to meet control criteria must be documented in a Nonconformance Memo (NCM). The NCM is automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group periodically reviews NCMs for potential trends. The NCM process is described in more detail in SOP DV-QA-0031. This is in addition to the corrective actions described in the following sections.

9.2 Batch Definition

Batches are defined at the sample preparation stage. The batch is a set of up to 20 samples of the same matrix, plus required QC samples (method blank, lab control sample, and matrix spike/matrix spike duplicate), processed using the same procedures and reagents within the same time period. Batches should be kept together through the whole analytical process as far as possible, but it is not mandatory to analyze prepared extracts on the same instrument or in the same sequence. A method blank must be run on each instrument. See Policy DV-QA-003P for further details.

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9.3 **Method Blanks**

For each batch of samples, analyze a method blank. The method blank is analyzed after the calibration standards, normally before any samples. For low-level volatiles in water, the method blank consists of reagent water. For low-level volatiles in soil, the blank medium is Ottawa sand. For medium-level volatiles, the method blank consists of 5.0 mL of methanol. Surrogates are added and the method blank is carried through the entire analytical procedure.

Acceptance Criteria: The method blank must not contain any analyte of interest at or above one-half the reporting limit (except common laboratory contaminants, see below) or at or above 5% of the measured concentration of that analyte in the associated samples, whichever is higher.

> The method blank must have acceptable surrogate recoveries. (See Section 9.4)

Corrective Actions:

If the analyte is a common laboratory contaminant (i.e., methylene chloride, acetone, 2-butanone), the data may be reported with qualifiers if the concentration of the analyte is less than five times the reporting limit. Such action must be taken in consultation with the client.

Reanalysis of samples associated with an unacceptable method blank is required when reportable concentrations are determined in the associated samples.

If there is no target analyte greater than one-half the RL in the samples associated with an unacceptable method blank, the data may be reported with qualifiers. Such action should be taken in consultation with the client.

If surrogate recoveries in the blank are not acceptable, the data must be evaluated to determine if the method blank has served the purpose of demonstrating that the analysis is free of contamination. If surrogate recoveries are low and there are reportable analytes in the associated samples, re-extraction of the blank and affected samples will normally be required. Consultation with the client should take place.

If reanalysis of the batch is not possible due to limited sample volume or other constraints, the method blank is reported, all associated samples are flagged with a "B", and appropriate comments may be made in the narrative to provide further documentation.

9.4 **Surrogates**

Every sample, blank, and QC sample is spiked with surrogates. Surrogate recoveries in samples, blanks, and QC samples must be assessed to ensure that recoveries are within

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established limits. The compounds included in the surrogate spiking solutions are listed in Tables 8 and 8A.

Acceptance Criteria: Acceptance limits for surrogate recoveries are set at \pm 3 standard

deviations around the historical mean. Surrogate recovery limits

are updated semi-annually and stored in the LIMS.

Corrective Actions: If any surrogates are outside limits, the following corrective actions must take place (except for dilutions):

Check all calculations for error.

- Ensure that instrument performance is acceptable.
- Recalculate the data and/or reanalyze if either of the above checks reveal a problem.
- Re-prepare and reanalyze the sample or flag the data as "Estimated Concentration" if neither of the above resolves the problem.

The decision to reanalyze or flag the data should be made in consultation with the client. It is necessary to reprepare/reanalyze a sample only once to demonstrate that poor surrogate recovery is due to matrix effect, unless the analyst believes that the repeated out of control results are not due to matrix effect.

If the surrogates are out of control for the sample, matrix spike, and matrix spike duplicate, then matrix effect has been demonstrated for that sample and re-preparation/reanalysis is not necessary. If the sample is out of control and the MS and/or MSD is in control, then reanalysis or flagging of the data is required.

9.5 **Laboratory Control Samples (LCS)**

An LCS is analyzed for each batch. The LCS is analyzed after the calibration standard, and normally before any samples. The LCS spiking solution is prepared from a different source than are the calibration standards. The LCS contains a representative subset of the analytes of interest (See Table 9), and must contain the same analytes as the matrix spike. For low-level volatiles in water, the LCS matrix is reagent water. For low-level volatiles in soil, the LCS matrix is Ottawa sand.

Acceptance Criteria: The LCS recovery for the control analytes must be within established control limits. Unless otherwise specified in a reference method or project requirements, the control limits are set at \pm 3 standard deviations around the mean of the historical data. An LCS that is determined to be within acceptance criteria effectively demonstrates that the analytical system is in control and validates system performance for the samples in the associated batch. Recovery limits are updated semi-annually and stored in the LIMS

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If there are a large number of analytes in the LCS, then a specified number of results may fall beyond the LCS control limit (3 standard deviations), but within the marginal exceedance (ME) limits, which are set at \pm 4 standard deviations around the mean of historical data. Marginal exceedances are recognized and allowed by NELAC, AFCEE, and the DOE. DoD requires individual project approval for the use of marginal exceedances. The number of marginal exceedances is based on the number of analytes in the LCS, as shown in the following table:

# of Analytes in LCS	# of Allowed Marginal Exceedances
> 90	5
71 – 90	4
51 – 70	3
31 – 50	2
11 – 30	1
< 11	0

If more analytes exceed the LCS control limits than is allowed, or if any analyte exceeds the ME limits, the LCS fails and corrective action is necessary. Marginal exceedances must be random. If the same analyte repeatedly fails the LCS control limits, it is an indication of a systematic problem. The source of the error must be identified and corrective action taken.

Note: Additional criteria are stated in the North Carolina QAS.

Note: Some programs (e.g., South Carolina) do not allow marginal exceedances. Please see the QSAS's in the public folders for the current requirements.

Corrective Actions:

If any analyte or surrogate is outside established control limits as described above, the system is out of control and corrective action must occur. Corrective action will normally be repreparation and reanalysis of the batch.

If the batch is not re-extracted and reanalyzed, the reasons for accepting the batch must be clearly presented in the project records and the report. Examples of acceptable reasons for not reanalyzing might be that the matrix spike and matrix spike duplicate are acceptable, and sample surrogate recoveries are good, demonstrating that the problem was confined to the LCS. This type of justification should be reviewed and documented with the client before reporting.

If re-extraction and reanalysis of the batch is not possible due to limited sample volume or other constraints, the LCS is reported,

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all associated samples are flagged, and appropriate comments are made in a narrative to provide further documentation.

9.6 Matrix Spike and Matrix Spike Duplicate (MS/MSD)

For each QC batch, analyze a matrix spike and matrix spike duplicate. compounds and levels are given in Table 9. The matrix spike/duplicate must be analyzed at the same dilution as the unspiked sample, even if the matrix spike compounds will be diluted out.

Acceptance Criteria: The MS/MSD recovery for the control analytes must be within established control limits. Unless otherwise specified in a reference method or project requirements, the control limits are set at \pm 3 standard deviations around the mean of the historical data. The relative percent difference (RPD) between the MS and the MSD must be less than the established RPD limit, which is based on statistical analysis of historical data. MS/MSD recovery and RPD limits are updated semi-annually and stored in the LIMS.

Corrective Actions:

If any individual recovery or RPD falls outside the acceptable range, corrective action must occur. The initial corrective action will be to check the recovery of that analyte in the LCS. Generally, if the recovery of the analyte in the LCS is within limits, then the laboratory operation is in control and analysis may proceed. The reasons for accepting the batch must be documented.

If the recovery for any component is outside QC limits for both the matrix spike/ spike duplicate and the LCS, the laboratory is out of control and corrective action must be taken. Corrective action will normally include reanalysis of the batch.

If an MS/MSD is not possible due to limited sample, then an LCS duplicate should be analyzed. The RPD between the LCS and LCSD is compared to the established acceptance limit.

9.7 **Acid Preservation or pH adjustment**

The stability of 2-chloroethylvinylether, acrolein, and according to the regulations. acrylonitrile is reduced when subjected to low pH. It is therefore not recommended that these compounds be analyzed routinely from preserved VOA vials and since there is no reasonable way to achieve pH between 4 and 5, it is recommended that unpreserved vials be used for the analysis of these compounds.

Acceptance Criteria: To ensure detection of these compounds, samples must be

processed correctly. Where Method 624 is being used for compliance purposes, the regulatory hold times take precedence.

Corrective Actions: If 624 data are not being generated for compliance purposes, the

technical stability of the compounds may be considered. Where

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method 8260 is the base method, it is allowable to qualify the results as estimated. To deviate from the regulatory hold times, the following documentation must be maintained:

- A NCM must be generated by the lab that the samples are for non-compliance.
- A NCM must be generated that results are not method compliant.

10.0 Procedure

- 10.1 One-time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using an NCM. The NCM is automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group periodically reviews NCMs for potential trends. The NCM process is described in more detail in SOP # DV-QA-0031. The NCM shall be filed in the project file and addressed in the case narrative.
- **10.2** Any deviations from this procedure identified after the work has been completed must be documented in an NCM, with a cause and corrective action described.
- 10.3 Sample Preservation using EnCore[™] Samplers.
 - 10.3.1 Preservation in Methanol (Medium-Level Analysis)
 - **10.3.1.1** Extrude the (nominal) 5 g sample from one of the Encore[™] samplers into a <u>tared</u> 20 mL VOA vial. Obtain the weight of the soil added to the vial and record it on the label. Quickly add 5 mL of methanol and cap the vial.
 - **10.3.1.2** If sufficient samplers are provided (or for the sample(s) designated by the client), prepare MS and MSD samples as above.
 - 10.3.1.3 Prepare a method blank and LCS sample by weighing approximately 5 g of baked Ottawa sand for each into separate, tared 20 mL VOA vials. Add 5 mL of methanol to the blank. For the LCS, the volume of methanol added is dependent upon the spike list. Add 4.95 mL methanol if the Short List is to be spiked and 4.85 mL methanol if the full list is to be spiked. Cap tightly. Store with the samples.
 - **10.3.1.4** Store the samples and QC samples in the freezer until screening is performed. Surrogates and LCS/MS/MSD spikes are only added if it is determined the samples will be analyzed at the medium level.

10.3.2 Preservation in Water (Low-Level Analysis)

10.3.2.1 Extrude the (nominal) 5 g sample from one of the Encore[™] samplers into a tared 20 mL VOA vial. Obtain the weight of the soil added to

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the vial and record it on the label. Quickly add 5 mL of water and a magnetic stirrer. Cap the vial. Repeat for the remaining aliquot.

- **10.3.2.2** If requested by the client, 1 g of sodium bisulfate is added to the second sample preserved with water.
- **10.3.2.3** If sufficient samplers are provided for a sample in the batch, or for any samples identified by the client, prepare MS and MSD samples as above.
- 10.3.2.4 Prepare a method blank and LCS sample by weighing approximately 5 g of baked Ottawa sand for each into separate, tared 20 mL VOA vials. Add 5 mL of water to the blank. For the LCS, the volume of water added is dependent upon the spike list. Add 4.95 mL water if the Short List is to be spiked and 4.85 mL methanol if the full list is to be spiked. Add a magnetic stirrer. Cap tightly. Store with the samples.
- 10.3.2.5 Store the samples and QC samples in the freezer until screening is performed. Surrogates and LCS/MS/MSD spikes are only added if it is determined the samples will be analyzed at the medium level.
- **10.3.3** Screen the samples. (See Section 10.5.) If the screen indicates any samples will be analyzed as medium level, go to Section 10.5.1. If the screen indicates any samples will be analyzed as low level, go to Section 10.7.7.

10.4 Sample Storage for Field Preserved Samples

- **10.4.1** Obtain the weight of the soil added to each vial and record it in TALS.
 - 10.4.1.1 Prepare a method blank and LCS sample by weighing approximately 5 g of baked Ottawa sand for each into separate, tared 20 mL VOA vials for each analysis method (medium-level and low-level).
 - 10.4.1.1.1 For the medium level method add 5 mL of methanol to the blank. For the LCS, the volume of methanol added is dependent upon the spike list. Add 4.95 mL methanol if the Short List is to be spiked and 4.85 mL methanol if the full list is to be spiked. Cap tightly. Store with the samples.
 - **10.4.1.1.2** For the low level method add water instead of methanol using the same volumes as in Section 10.4.1.1.1.
 - **10.4.1.2** Store the samples and QC samples in the freezer until screening is performed. Surrogates and LCS/MS/MSD spikes are only added if it is determined the samples will be analyzed at the medium level.
- 10.4.2 Screen the samples. (See Section 10.5) If the screen indicates any samples will be analyzed as medium level, go to Section 10.7.5. If the screen indicates any samples will be analyzed as low level, go to Section 10.7.7.

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10.5 Sample Screening

10.5.1 Where possible, samples are screened by headspace or GC/MS off-tune analysis to determine the correct aliquot for analysis. See SOP DV-MS-0009. Alternatively, an appropriate aliquot can be determined from sample histories.

10.6 Sample Preparation for Medium-Level Analysis – Field or Lab Preserved

- 10.6.1 For each of the samples that are determined to be Medium-Level samples by the screening procedure, add the correct amount of surrogate spiking mixture for a final concentration of 2 μ g/mL. Example: 4 μ L of 2500 μ g/mL for a nominal 5 g sample or 20 μ g/mL for a nominal 25 g sample. Cap the sample vial. Surrogates are added to all QC samples as well as field samples.
- 10.6.2 Add the correct amount of matrix spiking solution to the matrix spike and matrix spike duplicate samples for a final concentration of 2 μ g/mL.
- 10.6.3 Add the correct amount of matrix spiking solution to the LCS sample for a final concentration of 2 μ g/mL. If 25 g samples are being used, adjust the proportions for the LCS accordingly.
- **10.6.4** Shake the samples for two minutes to distribute the methanol throughout the soil.
- **10.6.5** Centrifuge the samples to clarify the extract.
- **10.6.6** Remove a portion of methanol and store in a clean Teflon-capped vial with no headspace refrigerated at ≤ 6 °C until analysis. Duplicate aliquots of the methanol extract should be taken and stored.

10.7 Sample Analysis Procedure

- **10.7.1** All analysis conditions for samples must be the same as for the initial and continuing calibration standards (including purge volume, time and flow, desorb time and temperature, column temperatures, multiplier setting etc.).
- 10.7.2 All samples must be analyzed as part of a batch. The batch is a set of up to 20 samples of the same matrix processed using the same procedures and reagents within the same time period. The batch also must contain a method blank, an LCS, and a MS/MSD.
 - 10.7.2.1 If there is insufficient time in the 12-hour tune period to analyze 20 samples, the batch may be continued into the next tune period. The 12-hour tuning requirements in Section 10.7.12.3 and 12-hour continuing calibration requirements in 10.7.14 must still be met. However, if any re-tuning or recalibration of the instrument is necessary, or if a period of greater than 24 hours from the preceding BFB tune has passed, a new QC batch must be started. For highlevel soils the batch is defined at the sample preparation stage.
 - 10.7.2.2 Laboratory generated QC samples (Blank, LCS, MS/MSD) do not

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count towards the maximum 20 samples in a batch. Field QC samples are included in the batch count.

10.7.2.3 Any reruns must be part of a valid batch. If dilutions of a sample are analyzed in the same calibration event they do not count towards the maximum batch count. (See DV-QA-003P.)

10.7.3 Water Samples

- **10.7.3.1** Purge-and-trap units that sample from a VOA vial should be equipped with a module that automatically adds surrogate and internal standard solution to the sample prior to purging the sample.
- **10.7.3.2** All samples and standard solutions must be at ambient temperature before analysis.
 - **NOTE:** Aqueous samples with high amounts of sediment present in the vial may not be suitable for analysis on this instrumentation, or they may need to be analyzed as soils.
- 10.7.3.3 To transfer a sample from its original container, fill a gas-tight syringe with the sample and adjust the sample volume based on the requested method. Place the measured sample into a clean VOA vial.
 - 10.7.3.3.1 For Method 8260, 20 mL sample aliquots are used unless dilutions are performed. (See Section 10.7.4.) Sample aliquots are measured in 25 mL gas tight syringes. Separate syringes are used for each sample.
 - **10.7.3.3.2** For Method 624, 5 or 20 mL sample aliquots are used unless dilutions are performed. (See Section 10.7.4.). Sample aliquots are measured in 5 or 25 mL gas-tight syringes. Separate syringes are used for each sample.
- **10.7.4** Dilutions should be done just prior to the GC/MS analysis of the sample. Dilutions are made in volumetric flasks or in a Luerlok syringe.
 - **10.7.4.1** For dilutions of aqueous samples which require less than 1 mL of sample the sample volume is added to 20 mL of reagent water in a VOA vial or in a gas-tight syringe.
 - 10.7.4.2 For dilutions of aqueous samples which require more than 1 mL of sample, the volume of reagent water is adjusted so that the total volume of sample and reagent water is 20 mL. The dilution is made in the VOA vial by adding the appropriate amount of reagent water to the vial. The sample aliquot is then added to the closed vial by injecting below the surface of the water.
 - **10.7.4.3** If the dilution required would use less than 5 μL of sample, then serial dilutions must be made in volumetric flasks.

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- 10.7.4.4 Check and document the pH of the remaining sample. Document the pH value on the run log. If the pH is not as expected, based on the sample type and preservation, document in an NCM in the LIMS.
- **10.7.4.5** Sample remaining in the vial after sampling is no longer valid for further analysis. A fresh VOA vial must be used for further sample analysis.
- 10.7.4.6 For TCLP samples, use 2.0 mL of TCLP leachate and spike it with 2.5 μ L of the 40 μ g/mL TCLP spiking solution. Bring to a volume of 20 mL with reagent water.
- **10.7.4.7** Surrogates and internal standards are added to each sample at the instrument at the time of purging.
- 10.7.4.8 Calibration standards and spiking solutions are added to the CCVs, LCS and MS/MSD samples by the analyst prior to purging by inserting the syringe needle through the septum into the water. Surrogates and internal standards are added to these samples by the instrument.
- **10.7.4.9** Purge the sample for eleven minutes (the trap should be below 50 °C).
- **10.7.4.10** After purging is complete, desorb the sample, start the GC temperature program, and begin data acquisition. After desorption, bake the trap for 2-5 minutes to condition it for the next analysis. When the trap is cool, it is ready for the next sample.
- **10.7.4.11** Desorb time, bake time, and temperature are optimized for the type of trap in use. Some programs or clients have special requirements for the desorb time. Method 624 requires a 4 minute desorb time.

NOTE: The same conditions must be used for samples and standards.

10.7.4.12 If foaming of the sample occurs, reanalyze the sample with the addition of 1 μ L of an antifoaming agent such as Antifoam B (J. T. Baker). A method blank spiked with 1 μ L of the Antifoam B must also be analyzed with the sample. Document in an NCM.

10.7.5 Methanol Extracts of Soils

- 10.7.5.1 Rinse a gas-tight syringe with organic-free water. Fill the syringe with the same volume of organic-free water as used in the calibrations (typically 5 mL).
- 10.7.5.2 Add no more than 25 μ L of methanolic extract (from Section 10.3.1 or 10.5.1) to the syringe for each sample and QC sample. Add surrogates to each sample.

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10.7.5.3 Calibration standards and spiking solutions are added to the CCVs, LCS and MS/MSD samples by the analyst prior to purging by inserting the syringe through the septum of the vial.

- 10.7.5.4 If less than 5 μ L of methanolic extract is to be added to the water, dilute the methanolic extract such that a volume greater than 5 μ L will be added to the water in the syringe.
- **10.7.5.5** Only internal standards are added at the instrument for methanol extracts.
- **10.7.5.6** Load the sample onto the purge and trap device and analyze as for aqueous samples. (See Section 10.7.3.)

10.7.6 Liquid Wastes that are Soluble in Methanol and Insoluble in Water

- **10.7.6.1** Pipette 2 mL of the sample into a tared vial. Use a top-loading balance. Record the weight to the nearest 0.1 gram.
- **10.7.6.2** Quickly add 7 mL of methanol, then add 1 mL of surrogate spiking solution to bring the final volume to 10 mL. Cap the vial and shake for 2 minutes to mix thoroughly.
- **10.7.6.3** For an MS/MSD pair, add 6 mL of methanol to 2 mL of the sample in a tared vial. Add 1 mL of surrogate solution and 1 mL of matrix spike solution.
- **10.7.6.4** Prepare an LCS by adding 1 mL of surrogate solution and 1 mL of matrix spike solution to 8 mL of methanol.
- **10.7.6.5** Rinse a gas-tight syringe with organic-free water. Fill the syringe with the same volume of organic-free water as used in the calibrations.
- **10.7.6.6** Add no more than 25 μL of methanolic extract (Section 10.7.6.2) to the syringe. Add internal standard (if used).
- **10.7.6.7** Load the sample onto the purge and trap device and analyze as for aqueous samples using 5 mL reagent water.
- 10.7.6.8 If less than 5 μ L of methanolic extract is to be added to the water, dilute the methanolic extract such that a volume greater than 5 μ L will be added to the water in the syringe. (See Section 10.7.4.)

10.7.7 Low-Level Soil Sample Analysis following SW846 Method 5035

- 10.7.7.1 This technique is to be used when samples are collected utilizing SW-846 Method 5035. Pre-weighed vials are used to collect approximately a 5 gram aliquot of soil (see section 8.2).
- **10.7.7.2** Purge-and-trap units that sample from the VOA vial should be equipped with a module that automatically adds surrogate and internal standard solution to the sample prior to purging the sample.

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10.7.7.3 If the autosampler uses automatic IS/SS injection, no further preparation of the VOA vial is needed. Otherwise, the internal and surrogate standards must be added to the vial.

- **10.7.7.4** The autosampler will heat and stir each sample as it is purged.
- **10.7.7.5** If any target analytes exceed the calibration range, analysis of the methanol preserved sample must be performed.

10.7.8 Low-Level Solids Analysis When Field Samples are Provided in a Jar

- **NOTE:** This technique may seriously underestimate analyte concentration and must not be used except at specific client request for the purpose of comparability with previous data. It is no longer part of SW-846.
- 10.7.8.1 This method is based on purging a heated sediment/soil sample mixed with water and, if applicable, internal and matrix spiking standards. Analyze all reagent blanks and standards under the same conditions as the samples (e.g., heated). The calibration curve is also heated during analysis. Purge temperature is 40 °C.
- **10.7.8.2** Do not discard any supernatant liquids. Mix the contents of the container with a narrow metal spatula.
- 10.7.8.3 Weigh out 5 g (or other appropriate aliquot) of sample into a clean VOA vial. Record the weight to the nearest 0.1 g. If method sensitivity is demonstrated, a smaller aliquot may be used. Do not use aliquots less than 1.0 g. If the sample is contaminated with analytes such that a purge amount less than 1.0 g is appropriate, use the medium-level method described in Section 10.7.5 with preparation described in Section 10.5.1.
- 10.7.8.4 Rinse a 5 mL gas-tight syringe with organic-free water, and fill. Compress to 5 mL. Inject the spiked water into the VOA vial that contains the soil sample and add a stirring bar.
- **10.7.8.5** The above steps should be performed rapidly and without interruption to avoid loss of volatile organics.
- 10.7.8.6 Prepare a Method Blank and LCS using 5 g of Ottawa sand and 5 mL of water. Add a stirring bar to each. Prepare the MS/MSD (based on the sample requested by the client. The LCS spiking solution is added via a syringe inserted through the septum of the vial to the LCS and MS/MSD samples.
- **10.7.8.7** Low level soil samples may be analyzed with a 1 g aliquot in place of the 5 g aliquot, mixed with water. If higher dilutions are required, the methanol extract (medium level) will be analyzed.
- **10.7.8.8** Surrogate and internal standards are added automatically to all samples at the instrument.

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- **10.7.8.9** The autosampler will heat and stir each sample as it is purged.
- **10.7.8.10** Soil samples that have low internal standard recovery when analyzed (< 50%) should be reanalyzed once to confirm matrix effect.

10.7.9 Initial Review and Corrective Actions

- 10.7.9.1 If the retention time for any internal standard in the continuing calibration changes by more than 0.5 minute from the mid-level initial calibration standard, the chromatographic system must be inspected for malfunctions and corrected. Reanalysis of samples analyzed while the system was malfunctioning is required.
- 10.7.9.2 If the internal standard response in the continuing calibration is more than 200% or less than 50% of the response in the mid-level of the initial calibration standard, the chromatographic system must be inspected for malfunctions and corrected. Reanalysis of samples analyzed while the system was malfunctioning is required.

Sample internal standard areas are compared to the mid-point of the supplemental initial calibration internal standard areas. Responses from 50% to 200% are acceptable. If a sample fails to meet these internal standard criteria, further investigation is necessary. If the change in sensitivity is a matrix effect confined to an individual sample, reanalysis is not necessary. If the change in sensitivity is due to instrumental problems, all affected samples must be reanalyzed after the problem is corrected.

10.7.9.3 The surrogate standard recoveries are evaluated to ensure that they are within limits. Corrective action for surrogates out of control will normally be to reanalyze the affected samples. However, if the surrogate standard response is out high and there are no target analytes or tentatively identified compounds, reanalysis may not be necessary. Out of control surrogate standard response may be a matrix effect. It is only necessary to reanalyze a sample once to demonstrate matrix effect, but reanalysis at a dilution should be considered.

10.7.10 Dilutions

10.7.10.1 If the response for any compound exceeds the working range of the GC/MS system, a dilution of the sample or extract is prepared and analyzed. An appropriate dilution should be in the upper half of the calibration range. Samples may be screened to determine the appropriate dilution for the initial run. If the initial diluted run has no hits or hits below 20% of the calibration range and the matrix allows for analysis at a lesser dilution, then the sample must be reanalyzed at a dilution targeted to bring the largest hit above 50% of the calibration range.

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10.7.10.2 Guidance for Dilutions Due to Matrix

If the sample is initially run at a dilution and the baseline rise is less than half the height of the internal standards, or if individual non target peaks are less than twice the height of the internal standards, then the sample should be reanalyzed at a more concentrated dilution. This requirement is approximate and subject to analyst judgment.

10.7.10.3 Reporting Dilutions

The most concentrated dilution with no target compounds above the calibration range will be reported. Other dilutions will be reported only at client request.

10.7.11 Instrument Set-up

Prior to the analysis of samples and blanks, the GC/MS system must be tuned and calibrated. Tuning is accomplished by analyzing 4-bromofluorobenzene (BFB) to establish that the GC/MS system meets the standard mass spectral abundance criteria. The GC/MS system must be calibrated initially at a minimum of five concentrations to determine the linearity of the response utilizing target calibration standards. The calibration must be verified each twelve-hour time period for each GC/MS system. The use of separate calibrations is required for water and low soil matrices.

10.7.12 Recommended Instrument Conditions

10.7.12.1 General

Electron Energy:	70 volts (nominal)
Mass Range:	35–300 amu
Scan Time:	to give at least 5 scans/peak,
	≤ 2 second/scan
Injector Temperature:	200 – 250 °C
Source Temperature:	According to manufacturer's specifications
Transfer Line:	Temperature: 250 – 300 °C
Purge Flow:	40 mL/minute
Carrier Gas Flow:	1-15 mL/minute, dependent upon column specifications

10.7.12.2 Gas Chromatograph Suggested Temperature Program

The following temperature programs vary with the column type used.

BFB Analysis

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Initial Temperature: 150 °C

Initial Hold Time: 0.00 minutes 1st Temperature Program: 50.00 °C/minute

Final Temperature: 220 °C

Final Time: 4.00 minutes

2nd Temperature Program: OFF Post Temperature: 0 °C

Post Time: 0.00 minutes
Run Time: 5.40 minutes

Sample Analysis

Initial Temperature: 40 °C
Initial Hold Time: 4 minutes

1st Temperature Program: 8 °C/minute
Final Temperature: 184 °C

2nd Temperature Program: 40 °C/minute

Final Temperature: 240 °C Final Hold Time: 2.6 minutes

10.7.12.3 Instrument Tuning

Each GC/MS system must be hardware-tuned to meet the abundance criteria listed in Table 10 for a maximum of a 50 ng injection or purging of BFB. Analysis must not begin until these criteria are met. These criteria must be met for each twelve-hour time period. The twelve-hour time period begins at the moment of injection of BFB.

10.7.13 Initial Calibration

- **10.7.13.1** Detailed information regarding calibration models and calculations can be found in Corporate SOP CA-Q-S-005, *Calibration Curves* (*General*) and in the public folder *Arizona Calibration Training*.
- 10.7.13.2 A series of five or more initial calibration standards is prepared and analyzed for the target compounds and each surrogate compound. Certain analytes are prepared at higher concentrations due to poor purge performance. The following calibration curves are maintained. Calibration levels for each analyte are given in the stated tables. Other calibration levels and purge volumes may be used depending on the capabilities of the specific instrument or program requirements.

Initial Calibration by Matrix and Method

Method	Matrix	Purge Volume	Calibration Levels
624	Water	5 mL	Table A-3
8260	Water	20 mL	Tables 5 and 5A

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Method	Matrix	Purge Volume	Calibration Levels
8260	Soil (low level) 5 ml		Tables 4 and 4A
8260	Soil (Methanol Extract)	5 mL reagent water + 25 µL Methanol	Tables 6 and 6A
Alaska	Soil	See Appendix B	See Appendix B

- 10.7.13.3 Calibration levels below the reporting limit may be removed provided that there is a minimum of five calibration points for linear regression and six calibration points for second order calibration. The lowest standard used in the calibration must be at or below the TestAmerica reporting limit.
- **10.7.13.4** The same purge volume must be used for calibration and sample analysis, and the low level standard must be at or below the reporting limit.
- **10.7.13.5** It may be necessary to analyze more than one set of calibration standards to encompass all of the analytes required for some tests.
- 10.7.13.6 Internal standard calibration is used. The internal standards are listed in Tables 7 and 7A. Target compounds should reference the nearest internal standard. Each calibration standard is analyzed and the response factor (RF) for each compound is calculated using the area response of the characteristic ions against the concentration for each compound and internal standard. See Equation 1, Section 11.4.1, for calculation of response factor.

10.7.13.7 Evaluation of retention times

The relative retention time of each target analyte in each calibration standard should agree within 0.5 min.

- **10.7.13.8** The % RSD of each of the calibration check compounds (CCC) must be less than or equal to 30%. Refer to Table 12. See Table A-2 for Method 624 criteria.
- **10.7.13.9** The average RF must be calculated for each compound. A system performance check is made prior to using the calibration curve. The five system performance check compounds (SPCC) are checked for a minimum average response factor. Refer to Table 11 for the SPCC compounds and required minimum response factors.
- 10.7.13.10 If the software in use is capable of routinely reporting curve

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coefficients for data validation purposes and the necessary calibration reports can be generated, then the analyst should evaluate analytes with %RSD > 15% for calibration on a curve. If it appears that substantially better accuracy would be obtained using quantitation from a curve then the appropriate curve should be used for quantitation. The correlation coefficient (coefficient of determination for non-linear curves) must be ≥ 0.990 .

Note: Additional criteria are stated in the North Carolina QAS.

10.7.13.11 If the software in use is capable of routinely reporting curve coefficients for data, and if the average of all the %RSDs in the calibration is > 15%, then calibration on a curve must be used for all analytes with %RSD > 15%. The analyst should consider instrument maintenance to improve the linearity of response. Otherwise, the correlation coefficient, r (coefficient of determination, r^2 for non-linear curves) must be ≥ 0.990 .

Note: Some states (like Arizona) and federal programs do not allow the use of grand mean. Refer to the Arizona QAS and SOP DV-QA-024P.

- 10.7.13.12 Once the initial calibration has been evaluated and determined to be valid, the calibration must be verified with an Initial Calibration Verification (ICV) using a standard prepared from an alternate source. All compounds in the ICV must be <35 % drift when compared to the initial calibration, except poor performers (see Table 15) which must be <55% drift. The ICV is generally run at the same concentration as the level 5 standard. See Table A-2 for method 624 criteria.</p>
- **10.7.13.13** If time remains in the 12-hour period initiated by the BFB injection before the initial calibration, samples may be analyzed. Otherwise, proceed to continuing calibration, Section 10.7.14.
- 10.7.13.14 A separate five point calibration must be prepared for analysis of low-level soils. Low-level soils analysis requires the use of a closed vial autosampler. Each standard is prepared by spiking the methanol standard solution through the septum of a VOA vial containing 5 mL of water. The standards are heated to 40°C for purging. All low-level soil samples, standards, and blanks must also be heated to 40°C for purging. Methanol soil extracts should be analyzed using the methanol calibration curve.
- 10.7.13.15 Non-standard analytes are sometimes requested. For these analytes, it is acceptable to analyze a single standard at the reporting limit with each continuing calibration rather than a five point initial calibration. The primary ion for the single standard must generate a peak clearly visible over background noise (greater than three standard deviations at a minimum) and be free of spectral interferences. If the analyte is detected in any of the samples, a five point initial calibration must be generated and the sample(s)

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reanalyzed for quantitation. However, if the analyte is not detected, the non-detect may be reported and no further action is necessary. A footnote or narrative comment should describe the basis of the reported result.

10.7.14 Continuing Calibration

- **10.7.14.1** The initial calibration must be verified every twelve hours.
- 10.7.14.2 Continuing calibration begins with analysis of BFB as described in Section 10.7.12.3. If the system tune is acceptable, the continuing calibration standard(s) are analyzed. The level 4 calibration standard is used as the continuing calibration standard. See Table A-2 for method 624 criteria.
- **10.7.14.3** The RF data from the standards are compared with the initial five-point calibration to determine the percent drift of the CCC compounds. The calculation is given in equation 4, Section 11.4.4.
- **10.7.14.4** The % drift of the CCCs must be ≤ 20% for the continuing calibration to be valid. The SPCCs are also monitored. The SPCCs must meet the criteria described in Table 11. In addition, the % drift for most non-CCC analytes must be ≤ 35 %, and for poor performers ≤ 50 % (See Table 15), with allowance for up to six target analytes to have a % drift greater than the applicable limit. For agencies that require specific control limits for non-CCC compounds (i.e., State of Arizona) see Table 14. See Table A-2 for method 624 criteria.

Note: Additional criteria are stated in the North Carolina QAS.

- **10.7.14.4.1** If none of the CCCs are required analytes, project specific calibration specifications (which may include the use of the CCCs listed in Table 12) must be agreed to with the client.
- 10.7.14.4.2 Cyclohexanone is unstable in the calibration solution forming 1,1-dimethoxycyclohexane. No calibration criteria are applied to cyclohexanone and quantitation is tentative. Cyclohexanone is included on the Universal Treatment Standard and FO-39 regulatory lists.
- 10.7.14.5 The retention time of the internal standards in the continuing calibration standard cannot change by more than 30 seconds when compared to the most recent five-point calibration. The internal standard areas must not change by more than a factor of 2 (50 200 %) from the mid point standard of the most recent five-point calibration.
- **10.7.14.6** If the CCCs and/or the SPCCs do not meet the criteria in Sections 10.7.14.3 and 10.7.14.4, the system must be evaluated and corrective action must be taken. The BFB tune and continuing

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calibration must be acceptable before analysis begins. Extensive corrective action, such as a different type of column, will require a new initial calibration.

- 10.7.14.7 Once the above criteria have been met, sample analysis may begin. Initial calibration average RFs (or the calibration curve) will be used for sample quantitation, not the continuing calibration RFs. Analysis may proceed until 12 hours from the injection of the BFB have passed. (A sample desorbed less than or equal to 12 hours after the BFB is acceptable.)
- **10.7.14.8** Sodium Bisulfate must be added to the CCV when analyzing samples preserved with it.

11.0 Calculations / Data Reduction

11.1 Detailed calibration equations can be found in the corporate SOP CA-Q-S-005 "Calibration Curves" and in the public folder, *Arizona Calibration Training*.

11.2 Qualitative Identification

11.2.1 An analyte is identified by retention time and by comparison of the sample mass spectrum with the mass spectrum of a standard of the suspected compound (standard reference spectrum). Mass spectra for standard reference may be obtained on the user's GC/MS by analysis of the calibration standards or from the NIST Library (same library as used for routine sample analysis). Two criteria must be satisfied to verify identification: (1) elution of sample component at the same GC retention time as the standard component; and (2) correspondence of the sample component and the standard component characteristic ions.

NOTE: Care must be taken to ensure that spectral distortion due to co-elution is evaluated.

- 11.2.1.1 The sample component retention time must compare to within \pm 0.2 min. of the retention time of the standard component. For reference, the standard must be run within the same twelve hours as the sample.
- 11.2.1.2 All ions present in the standard mass spectra at a relative intensity greater than 10% (most abundant ion in the spectrum equals 100%) should be present in the sample spectrum.
- 11.2.1.3 The relative intensities of ions should agree to within ±30% between the standard and sample spectra. (Example: For an ion with an abundance of 50% in the standard spectra, the corresponding sample abundance must be between 20 and 80%.)
- 11.2.2 If a compound cannot be verified by all the above criteria, but in the technical judgment of the analyst, the identification is correct, then the analyst shall report that identification and proceed with quantitation.

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11.2.3 All data are subject to two levels of technical review, as described in SOP DV-QA-0020.

11.3 Tentatively Identified Compounds (TICs)

- 11.3.1 If the client requests components not associated with the calibration standards, a search of the NIST library may be made for the purpose of tentative identification. The following guidelines apply:
 - 11.3.1.1 Relative intensities of major ions in the reference spectrum (ions > 10% of the most abundant ion) should be present in the sample spectrum.
 - 11.3.1.2 The relative intensities of the major ions should agree to within 20%. (Example: If an ion shows an abundance of 50% in the standard spectrum, the corresponding sample ion abundance must be between 30% and 70%).
 - **11.3.1.3** Molecular ions present in the reference spectrum should be present in the sample spectrum.
 - 11.3.1.4 Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or presence of co-eluting compounds.
 - 11.3.1.5 Ions present in the reference spectrum but not in the sample spectrum should be reviewed for possible subtraction from the spectrum because of background contamination or co-eluting peaks. (Data system reduction programs can sometimes create these discrepancies.)
 - 11.3.1.6 Computer-generated library search routines should not use normalization routines that would misrepresent the library or unknown spectra when compared to each other. Only after visual inspection of the sample with the nearest library searches should the analyst assign a tentative identification.

11.4 Calculations.

11.4.1 Response factor (RF):

$$RF = \frac{A_x C_{is}}{A_{is} C_x}$$
 Equation 1

Where:

 A_x = Area of the characteristic ion for the compound to be measured.

 A_{is} = Area of the characteristic ion for the specific internal standard.

 C_{is} = Concentration of the specific internal standard, ng

 C_x = Concentration of the compound being measured, ng.

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11.4.2 Standard deviation (SD):

$$SD = \sqrt{\frac{\sum_{i=1}^{n} (X_i - \overline{X})^2}{n-1}}$$
 Equation 2

Where:

 X_i = Value of X at i through n. \underline{n} = Number of points. \overline{X} = Average value of X_i .

11.4.3 Percent relative standard deviation (%RSD):

$$\% RSD = \frac{SD}{RF} \times 100\%$$
 Equation 3

Where \overline{RF} is the mean of RF values for the calibration.

11.4.4 Percent drift between the initial calibration and the continuing calibration:

$$\% Drift = \frac{C_{\text{expected}} - C_{found}}{C_{\text{expected}}} \times 100\%$$
 Equation 4

Where:

 $C_{\text{expected}} = Known concentration in standard.$ $C_{\text{found}} = Measured concentration using selected quantitation method.}$

- **11.4.5** See SOP CA-Q-S-005 for more detailed calibration equations.
- **11.4.6** Target compound and surrogate concentrations:

Concentrations in the sample may be determined from linear or second order (quadratic) curve fitted to the initial calibration points, or from the average response factor of the initial calibration points. Average response factor may only be used when the % RSD of the response factors in the initial calibration is \le 15%.

11.4.6.1 Calculation of concentration using Average Response Factors:

Concentration
$$(\mu g/L) = \frac{x}{RF}$$
 Equation 5

11.4.6.2 Calculation of concentration using Linear fit:

Concentration (
$$\mu$$
g/L) = $A + Bx$ Equation 6

11.4.6.3 Calculation of concentration using Quadratic fit:

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Concentration($\mu g/L$) = $A + Bx + Cx^2$

Equation 7

Where:

x = see equations 8, 9, and 10.

A = intercept of the calibration function.

B = slope of calibration function.

C = curvature of a second-order calibration function.

11.4.6.4 Calculation of x for Water and water-miscible waste:

$$x = \frac{A_x I_s D_f}{A_{is} V_0}$$
 Equation 8

Where:

= Area of characteristic ion for the compound being measured (secondary ion quantitation is allowed only when there are sample interferences with the primary ion).

 A_{is} = Area of the characteristic ion for the internal standard.

 I_s = Amount of internal standard added in ng.

 $D_f = \frac{\text{Total volume purged (mL)}}{\text{Volume of original sample used (mL)}}$

 $V_o = Volume of water purged, mL.$

11.4.6.5 Calculation of x for High-level soils:

$$x = \frac{(A_x)(I_s)(V_t)(1000)D_f}{(A_{is})(V_a)(W_s)(D)}$$
 Equation 9

Where:

 A_x , I_s , D_f , A_{is} , = same as used in equation 8 above. V_t = Volume of total extract, mL (typically 25 mL). V_a = Volume of extract added for purging, $\underline{\nu}$ L. W_s = Weight of sample extracted, g.

100 – % moisture D 100

11.4.6.6 Calculation of x for Low level soils:

$$x = \frac{(A_x)(I_s)}{(A_{is})(W_s)(D)}$$
 Equation 10

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Where:

 A_x , I_s , D_f , A_{is} , = same as used in equation 8 above. D = same as in equation 9 above. W_s = Weight of sample added to the purge vessel, g.

11.4.6.7 Calculation of TICs

The calculation of TICs (tentatively identified compounds) is identical to the above calculations with the following exceptions:

 A_x = Area in the total ion chromatogram for the compound being measured.

 A_{is} = Area of the total ion chromatogram for the nearest internal standard without interference.

RF = 1

In other words, the concentration is equal to x as defined in equations 8, 9, and 10.

11.4.7 MS/MSD Recovery

$$\% \text{Recovery} = \frac{SSR - SR}{SA} \times 100\%$$
 Equation 11

Where:

SSR = Spike sample result. SR = Sample result. SA = Spike added.

11.4.8 Relative % Difference calculation for the MS/MSD:

$$RPD = \frac{\left| MSR - MSDR \right|}{\frac{1}{2} \left(MSR + MSDR \right)} \times 100\%$$
 Equation 12

Where:

RPD = Relative percent difference.

MSR = Matrix spike result.

MSDR = Matrix spike duplicate result.

12.0 <u>Method Performance</u>

12.1 Method Detection Limit Study (MDL)

12.1.1 The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL procedure in TestAmerica Denver's Policy No. DV-QA-005P. MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method or program requirements (e.g., DoD) indicate a greater frequency.

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12.2 Demonstration of Capabilities

12.2.1 All personnel are required to perform an initial demonstration of proficiency (IDOC) on the instrument they will be using for analysis prior to testing samples. On-going proficiency must be demonstrated annually. IDOCs and on-going proficiency demonstrations are conducted as follows.

- **12.2.1.1** Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation. The concentration of the QC check sample should be equivalent to a mid-level calibration.
- **12.2.1.2** Calculate the average recovery and standard deviation of the recovery for each analyte of interest.
- 12.2.1.3 If any analyte does not meet the acceptance criteria, the test must be repeated. Only those analytes that did not meet criteria in the first test need to be evaluated. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.
- **12.2.1.4** Further details concerning demonstrations of proficiency are described in SOP DV-QA-0024.

12.3 Training Requirements

- 12.3.1 The Group Leader is responsible for ensuring that this procedure is performed by an associate who has been properly trained in its use and has the required experience. See requirements for demonstration of analyst proficiency in SOP DV-QA-0024.
- 12.3.2 Each analyst performing the method must complete a demonstration of capability (DOC) by successfully preparing and/or analyzing four consecutive LCSs, or a blind performance evaluation (PE) sample, or other acceptable QC samples. The results of the DOC study are summarized in the NELAC format, as described in SOP DV-QA-0024. DOCs are approved by the Quality Assurance Manager and the Technical Director. DOC records are maintained by the QA staff in the central training files. Analysts who continue to perform the method must successfully complete a demonstration of capability annually.

13.0 Pollution Control

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability).

14.0 Waste Management

14.1 All waste will be disposed of in accordance with Federal, State, and local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this procedure, the

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policies in section 13, "Waste Management and Pollution Prevention", of the Environmental Health and Safety Manual, and DV-HS-001P, "Waste Management Program."

- **14.2** The following waste streams are produced when this method is carried out:
 - 14.2.1 Methanol Waste Vial Waste and Flammable Waste Streams A and C
 - **14.2.2** Expired Chemicals/Reagents/Standards Contact Waste Coordinator
 - 14.2.3 Acidified Water Waste Stream W
 - **NOTE:** Radioactive waste, mixed waste, and potentially radioactive waste must be segregated from non-radioactive waste as appropriate. Contact the Radioactive Waste Coordinator for proper management of these materials.

15.0 References / Cross-References

- 15.1 SW-846, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, Third Edition and all promulgated updates, EPA Office of Solid Waste, January 2005.
 - **15.1.1** Method 8260B, Volatile Compounds by Gas Chromatography/Mass Spectrometry (GC/MS), Revision 2, December, 1996.
 - **15.1.2** Method 5030B, Purge-and-Trap for Aqueous Samples, Revision 2, December, 1996.
 - **15.1.3** Method 5035, Closed-System Purge-and-Trap and Extraction for Volatile Organics in Soil and Waste Samples, Revision 0, December, 1996.
 - **15.1.4** Method 5035A (R1-MIR), Closed-System Purge-and-Trap and Extraction for Volatile Organics in Soil and Waste Samples, Draft Revision 1, July 2002.
 - **15.1.5** Method 8000B, Determinative Chromatographic Separations, Revision 2, December 1996.
 - **15.1.6** Method 8000C, Determinative Chromatographic Separations, Revision 3, March 2003...
- **15.2** 40 CFR Part 136, Appendix A (Method 624, Method 603).
- **15.3** Method AK101 For the Determination of Gasoline Range Organics, Alaska DEC, Version 04/08/02.

16.0 <u>Method Modifications:</u>

Item	Method	Modification
1	SW-846 8260B	lon 119 is used as the quantitation ion for chlorobenzene-d ₅ .
2	SW-846 8260B	The quantitation and qualifier ions for some compounds have been

Item	Method	Modification	
		changed from those recommended in SW-846 in order to improve the reliability of qualitative identification.	
3	SW-846 8260B	This SOP has been written to allow for a 20 mL purge volume for waters. An additional 5 mL of DI water is added to all samples, QC and calibration standards. The final purge volume is 25 mL.	
4	SW-846 8260B	Method 8260B recommends that the purge vessel is run through an additional purge cycle after 25 mL sample analysis to remove carryover. Instead, purge vessels are oven baked between analyses or disposable vessels are used one time only.	
5	SW-846 8260B	SW-846 recommends that a curve be used for any analytes with %RSD of the response factors > 15%. However, some industry standard data systems and forms generation software cannot report this data with the necessary information for data validation. In addition, most software available does not allow weighting of the curve. Unweighted curves may exhibit serious errors in quantitation at the low end, resulting in possible false positives or false negatives. Therefore, if the overall average is < 15% then the ICAL is considered acceptable and any compounds that are not <15% will use linear regression.	
6	EPA 624	Method 624 is required for demonstration of compliance with CWA permits, e.g., NPDES wastewater discharge permits. This method can be applied only to aqueous matrices. The standard analyte list and reporting limits are listed in Table A-1. If compounds are added to the analysis, all of the method criteria must be satisfied for the additional compounds.	
7	EPA 624	The tune period for this method is defined as 24 hours, which is the maximum elapsed time before the tune check is performed. Calibration verifications are done at the same 24 hour frequency.	
8	EPA 624	The initial calibration curve for this method requires at least three points, as shown in Table A-3.	
9	EPA 624	Sample concentrations are calculated using the average RRF from the initial calibration curve.	
10	EPA 624	Each target analyte is assigned to the closest eluting internal standard.	
11	EPA 624	 Initial demonstration of Proficiency The spiking level for the four replicate initial demonstration of proficiency is 20 μg/L. The acceptance criteria are listed in Table A-2 	
12	EPA 624	 Initial calibration curve requirements: Target compounds must have RSD ≤ 35%. 	

Item	Method	Modification
		If this requirement can not be met, a regression curve must be constructed for the non-compliant compounds. There is no correlation coefficient requirement for the regression curve.
		Continuing calibration verification requirements:
13	EPA 624	The continuing calibration standard is from a different source than the initial calibration standard. The daily CCAL concentration is 20 ug/L. The acceptance criteria are listed in Table A-2.
	L17(024	Matrix Spike and LCS Requirements
		 The matrix spike and LCS are spiked at 20 µg/L, prepared from the same source containing all analytes of interest. A matrix spike duplicate is not necessary for this method. The recovery limits for matrix spike and LCS recovery are listed in Table A-2.
14	EPA 624	Consistent with the other volatile methods, corrections for recovery are not allowed.
15	EPA 624	Qualitative Identification – The source method states that the relative intensities of ions should agree to within ±20% between the standard and sample spectra. This SOP uses ±30%. (Example: For an ion with an abundance of 50% in the standard spectra, the corresponding sample abundance must be between 20 and 80 percent.)
16	EPA 624	Section 5.2.2 of the source method describes the trap packing materials as Tenax GC, Methyl silicone, silica gel and coconut charcoal. TestAmerica routinely employs the OI #10 trap which consists of Tenax/Silica Gel/ Carbon Molecular Sieve or the Supelco Vocarb 3000 which consists of Carbopack B, Carbonxen1000 and 1001.
17	EPA 624	Section 5.3.2 of the source method describes a packed analytical column. TestAmerica routinely employs capillary columns when performing this method.
18	EPA 624	The source method provides a suggested list of compounds for internal and surrogate standards. Others are permitted by the method. TestAmerica uses three internal standards, including chlorobenzene-d ₅ and 1,4-dichlorobenzene-d ₄ , which are not listed in Table 3 of the source method. Toluene-d ₈ is used as a surrogate compound, which is also not listed in the source method.
19	EPA 624	The lab is preparing internal standards at 10 ug/L and applying the same criteria designed for 30 ug/L in the Method. The lower concentration is consistent with the greater sensitivity provided by capillary columns as compared to the older packed columns described in the method. It could only be more challenging for the lab to meet the acceptance criteria at 10 ug/L; it provides a higher

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Item	Method	Modification	
		level of data quality.	
20	EPA 624	Method 624 describes a mass scan range of 25 to 260 amu. Table 13 lists all of the ions used for analysis. None of the ions are below 35 amu. Therefore, we scan from 35 to 300 and include all ions needed for analysis.	
21	EPA 624	Method 624 describes dilutions "if response of any m/z" exceeds the response for the highest m/z in the ICAL. As the m/z ratio is always directly proportional to the concentration, evaluation based on dilution (per 11.10) is equivalent.	
22	EPA 624	Method 624 has criteria for unresolved isomers. The problems of isomeric resolution for the routine analytes listed in this SOP were worked through when the laboratory developed its implementation of the method. For example, we know through experience that meta- and para-xylenes will not be resolved and it was not necessary to include an evaluation for the xylenes in each analysis. meta- and para-xylenes are reported as an isomeric pair. Any development work to add compounds would take this into account.	
23	624	The source method recommends Method 603 as the preferred method for Acrolein and Acrylonitrile. Method 624 is recommended as a screening method (see section 1.2 of Methods 603 and 624). Calibration and quality control samples indicate that the conditions described in this SOP are suitable for the analysis of Acrolein and Acrylonitrile. EPA's Method Update Rule (MUR), May 18, 2012, allows the addition of acrolein and acrylonitrile to Method 624, using the preservation, holding time and QC acceptance criteria from Method 603. As states implement the MUR Method 624 becomes a determinative method for these two analytes. Until such time, Method 624 remains a screening method for regulatory compliance.	
24	SW846 5035	The source method recommends adding approximately the same amount of the sodium bisulfate preservative as the sample (e.g., ~ 1 g), as the presence of the preservative will affect the purging efficiencies of the analytes. TestAmerica Denver does not recommend the use of sodium bisulfate to preserve soil samples, but encourages clients to collect samples using other available methods. The use of this preservative has been shown to cause difficulties recovering more reactive analytes on the purge and trap system (e.g. 2-Chloroethyl vinyl ether, acrylamide).	

17.0 Attachments

Table 1.	TestAmerica Primary List Reporting Limits for 8260B
Table 2.	TestAmerica 8260 Secondary List Reporting Limits
Table 3.	TestAmerica Appendix IX List Reporting Limits
Table 4.	Soil Calibration Levels, 5-gram Purge (μg/Kg)
	(Standard Mixes: MV-Main & MV-Main GasKe)

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Table 4A.	Soil Calibration Levels, 5 gram Purge (μg/Kg)	
	(Standards: MV-Supp Std and MV-2 Cleve)	
Table 5.	Water 8260 List Calibration Levels (μg/L)	
	(Standards: MV-Main and MV-Main GasKe)	
Table 5A	Water 8260 List Calibration Levels (μg/L)	
	(Standards: MV-Supp Std and MV- 2 Cleve)	
Table 6.	Medium Level Soil 8260 List Calibration Levels (μg/Kg)	
	(Standards: MV-Main and MV-Main GasKe)	
Table 6A.	Medium Level Soil 8260 List Calibration Levels (μg/Kg)	
	(Standards: MV-Supp Std and MV- 2 Cleve)	
Table7.	Manually added Internal Standards	
Table 7A.	Automatically Added Internal Standards	
Table 8.	Manually Added Surrogate Standards	
Table 8A.	Automatically Added Surrogate Standards	
Table 9.	Matrix Spike and LCS Standard	
Table 10.	BFB Key Ion Abundance Criteria	
Table 11.	SPCC Compounds and Minimum Response Factors	
Table 12.	CCC Compounds	
Table 13.	Characteristic Ions	
Table 14.	State of Arizona ICV/CCV Quality Control Limits	
Table 15.	List 1 Poorly performing Compounds	
Table A-1.	Method 624 Analytes and Reporting Limits, 5-mL Purge	
Table A-2.	Method 624 QC Acceptance Criteria	
Table A-3.	Calibration Levels for 624, 5 mL Purge	
Appendix A.	Modifications for Analysis of 1,4-Dioxane, 1,2,3-Ttrichloropropane, 1	,2-
• •	Dibromo-3-chloropropane, and 1,2-Dibromoethane by Selected I	on
	Monitoring	
Table Ap-1.	TAL Method 8260SIM Standard Reporting Limits	
Table Ap-2.	Method 8260SIM Calibration Levels	
Table Ap-3.	Method 8260SIM LCS Spike Concentrations	
Table Ap-4.	8260SIM Surrogate Compounds	
Table Ap-5.	8260SIM Internal Standard Compounds	
Table Ap-6.	8260 Selected Masses	
Table Ap-7.	Suggested Instrument Conditions for 8260SIM	
Appendix B	Modifications for Analysis of Soils Collected for the State of Alaska	
Table Bp-1:	TestAmerica 8260 Reporting Limits – AK Soils	
Table Bp-2:	Calibration Levels for 8260, 5035FM_AK	
Table Bp-3:	5035FM_AK Calibration Levels (μg/Kg)	
	(Standards: MV-Supp Std and MV-2 Cleve)	
Attachmant 1	Con Standarda Tradking Lag	

18.0 Changes from Previous Revision

- Revision 7, dated 27 July 2012
 - Added sodium bisulfate to Section 7.

Attachment 1. Gas Standards Tracking Log

 Revised Section 8 to include Terra Core samplers and moved instructions on sample preparation and handling in the lab to Section 10. Reorganized sampling and preservation information into tables. Updated information including footnote

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on Holding Time and preservation table for water regarding Method Update Rule that approves use of Method 624 for analysis of acrolein and acrylonitrile.

- Removed flowcharts from Section 8.
- Revised Section 9.1
- Revised Section 10.
- Updated reference section to include Method 603, Method 5035A, and Method 8000B and 8000C.
- Revised Method Modifications #23
- Updated tables to reflect current practice.
- Added Appendix B for the analysis of soils using the AK methanol extraction procedure.
- Formatting and editorial changes throughout
- Revision 6.4, dated 28 December 2011
 - Changed the column ID and film thickness in section 6.1.8.1
 - Updated the calibration levels in Table AP-2
- Revision 6.3, dated 26 October 2011
 - Added Section 4.6 regarding interferences with toluene-d₈ surrogate when potassium permanganate may have been added to sample
 - o Updated path to QAS folders in the public folders, section 9.7
 - o Added J. T. Baker Antifoam B and reagent sand, sections 7.3, 7.4
 - Added description of procedure for use of antifoaming agent B, section 10.1.3.8
 - Formatting
- Revision 6.2, dated 25 August, 2011
 - Added requirements to section 9.4 for the use of Ottawa sand in soil LCS's.
- Revision 6.1, dated 31 January, 2011
 - Added details to Appendix A for the analysis of soils by SIM
 - o Added Tables AP-1 through Ap-7
 - o Added Attachment 1, Gas Standards Tracking Log
 - Added section 11.1 referencing corporate SOP CA-Q-S-005 "Calibration Curves"
- Revision 6, dated 02 November, 2010
 - Added analysis information concerning BFB
- Revision 4, dated May 5, 2010
 - Updated Tables to reflect current report limits.
 - Updated low level procedure to include water option for preservation.
 - Updated surrogate and spike amounts.
- Revision 3.1, dated 11 December 2009
 - o Added Trichloroethene to Table 11.
 - Updated section 16 to describe the process of adding and additional 5 mL of DI water to all samples and QC.
 - Added a note to section 9.4 that marginal exceedances are not allowed for some programs.
 - Updated the language in section 16 item 5 to describe the current practice.
- Revision 3.0, dated 21 January 2009

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- o Added clarification of sample preservation requirements to section 8.
- o Adjusted Table 16 for South Carolina requirements to utilize default limits.
- Added Table 8A for AFCEE water calibration levels.
- Revision 2.1, dated 16 July 2007
 - Add reference to North Carolina QAS for additional requirements to sections 9.6, 10.4.8, and 10.5.4.
 - o Remove Nitrogen as an allowable substitution for Helium in section 6.8.
 - o Added the current list of spike compounds to Table 11.
 - Updated references to include 5030B and 5035.
 - o Removed EPA 524.2 references.
- Revision 2.0
 - The method blank acceptance criteria and corrective actions were updated in Section 9.4.

Table 1. TestAmerica Primary List Reporting Limits for 8260B

		Reporting Limits ¹		
Compound	CAS Number	20 mL Water(µg/L)	Low Soil (µg/kg)	Med Soil (µg/kg)
Dichlorodifluoromethane	75-71-8	2	10	500
Chloromethane	74-87-3	2	10	500
Bromomethane	74-83-9	2	10	500
Vinyl chloride	75-01-4	1	5	500
Chloroethane	75-00-3	2	10	500
Trichlorofluoromethane	75-69-4	2	10	500
Acrolein	107-02-8	20	50	5,000
Acetone	67-64-1	10	20	1,000
Trichlorotrifluoroethane	76-13-1	3	20	1,000
Ethanol	64-17-5	300	600	10,000
Iodomethane	74-88-4	1	5	250
Carbon disulfide	75-15-0	2	5	250
Methylene chloride	75-09-2	2	5	250
tert-Butyl alcohol	75-65-0	50	200	10,000
1,1-Dichloroethene	75-35-4	1	5	250
1,1-Dichloroethane	75-34-3	1	5	250
trans-1,2-Dichloroethene	156-60-5	1	2.5	125
Acrylonitrile	107-13-1	20	50	5,000
Methyl <i>tert</i> -butyl ether (MTBE)	1634-04-4	5	20	250
Hexane	110-54-3	2	5	250
cis-1,2-Dichloroethene	156-59-2	1	2.5	125
1,2-Dichloroethene (Total)	540-59-0	1	5	250
Tetrahydrofuran	109-99-9	7	20	1,000
Chloroform	67-66-3	1	10	250
1,2-Dichloroethane	107-06-2	1	5	250
Dibromomethane	74-95-3	1	5	250
2-Butanone	78-93-3	6	20	1,000
1,4-Dioxane	123-91-1	200	500	25,000
1,1,1-Trichloroethane	71-55-6	1	5	250
Carbon tetrachloride	56-23-5	1	5	250
Bromodichloromethane	75-27-4	1	5	250
1,2-Dichloropropane	78-87-5	1	5	250

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Table 1. TestAmerica Primary List Reporting Limits for 8260B

		Reporting Limits ¹		
Compound	CAS Number	20 mL Water(µg/L)	Low Soil (µg/kg)	Med Soil (μg/kg)
cis-1,3-Dichloropropene	10061-01-5	1	5	250
Trichloroethene	79-01-6	1	5	250
Dibromochloromethane	124-48-1	1	5	250
1,2-Dibromoethane	106-93-4	1	5	250
1,2,3-Trichloropropane	96-18-4	2.5	5	250
1,1,2-Trichloroethane	79-00-5	1	5	250
Benzene	71-43-2	1	5	250
Ethylmethacrylate	97-63-2	3	5	250
trans-1,3-Dichloropropene	10061-02-6	3	5	250
Bromoform	75-25-2	1	5	250
4-Methyl-2-pentanone	108-10-1	5	20	1,000
2-Hexanone	591-78-6	5	20	1,000
Tetrachloroethene	127-18-4	1	5	250
Toluene	108-88-3	1	5	250
1,1,2,2-Tetrachloroethane	79-34-5	1	5	250
2-Chloroethyl vinyl ether ²	110-75-8	N/A ²	50	2,500
Vinyl acetate	108-05-4	3	10	500
Chlorobenzene	108-90-7	1	5	250
Ethylbenzene	100-41-4	1	5	250
Styrene	100-42-5	1	5	250
trans-1,4-Dichloro-2-butene	110-57-6	3	5	250
m- and p-Xylenes	179601-23-1	2	3.5	250
o-xylene	95-47-6	1	2.5	125
Total xylenes	1330-20-7	2	10	250
1,3-Dichlorobenzene	541-73-1	1	5	250
1,4-Dichlorobenzene	106-46-7	1	5	250
1,2-Dichlorobenzene	95-50-1	1	5	250

Reporting limits listed for soil/sediment are based on wet weight. The reporting limits calculated by the laboratory for soil/sediment, calculated on dry weight basis, will be higher.

² 2-Chloroethyl vinyl ether cannot be reliably recovered from acid preserved samples

Table 2. TestAmerica 8260 Secondary List Reporting Limits

Compound	CAS Number	Reporting Limits ¹		
		20 mL Water μg/L	Low Soil µg/kg	Medium Soil μg/kg
2,2-Dichloropropane	590-20-7	1	5	250
Bromochloromethane	74-97-5	1	5	250
1,1-Dichloropropene	563-58-6	1	5	250
1,3-Dichloropropane	142-28-9	1	5	250
1-Chlorohexane	544-10-5	1	5	500
1,1,1,2-Tetrachloroethane	630-20-6	1	5	250
Isopropylbenzene	98-82-8	1	5	250
Bromobenzene	108-86-1	1	5	250
n-Propylbenzene	103-65-1	1	5	250
2-Chlorotoluene	95-49-8	1	5	250
4-Chlorotoluene	106-43-4	1	5	250
1,3,5-Trimethylbenzene	108-67-8	1	5	250
tert-Butylbenzene	98-06-6	1	5	250
1,2,4-Trimethylbenzene	95-63-6	1	5	250
sec-Butylbenzene	135-98-8	1	5	250
4-Isopropyltoluene	99-87-6	1	5	250
n-Butylbenzene	104-51-8	1	5	250
1,2-Dibromo-3-chloropropane	96-12-8	5	5	250
1,2,4-Trichlorobenzene	120-82-1	1	5	250
Naphthalene	91-20-3	1	5	500
Hexachlorobutadiene	87-68-3	1	5	250
1,2,3-Trichlorobenzene	87-61-6	1	5	250
2-Pentanone	107-87-9	5	10	500
cis-1,4-Dichloro-2-butene	1476-11-5	3	5	250
Ethylene oxide	75-21-8	600	3,000	150,000

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Table 3. TestAmerica Appendix IX List Reporting Limits

		I	Reporting Limits	1
Compound	CAS Number	20 mL Water μg/L	Low Soil μg/kg	Medium Soil μg/kg
Allyl Chloride	107-05-1	2	10	500
Acetonitrile	75-05-8	30	100	5,000
Dichlorofluoromethane	75-43-4	2	10	25,000
Isopropyl ether	108-20-3	10	50	2,500
Chloroprene	126-99-8	1	5	500
n-Butanol	71-36-3	60	200	10,000
Propionitrile	107-12-0	20	50	1,000
Methacrylonitrile	126-98-7	10	50	2,500
Isobutanol	78-83-1	110	200	10,000
Methyl methacrylate	80-62-6	4	5	250
1,1,1,2-Tetrachloroethane	630-20-6	1	5	250
1,2-Dibromo-3-chloropropane	96-12-8	5	10	500
Ethyl ether	60-29-7	2	10	500
Ethyl Acetate	141-78-6	5	10	500
2-Nitropropane	79-46-9	5	10	500
Cyclohexanone ²	108-94-1	N/A ²	N/A ²	N/A ²
Isopropylbenzene	98-82-8	1	5	250

¹ Reporting limits listed for soil/sediment are based on wet weight. The reporting limits calculated by the laboratory for soil/sediment, calculated on dry weight basis, will be higher.

Cyclohexanone decomposes to 1,1-dimethoxycyclohexane in methanolic solution. Reporting limits cannot be accurately determined.

Table 4. Soil Calibration Levels, 5-gram Purge¹ (Standard Mixes: MV-Main & MV-Main GasKe)

	allualu N				Level, µg/	-		
Compound	Lovel	Level	Level	Level	Level	Level	Level	Level
	Level 1	2	3	4	5	6	7	8
1,1,1,2-Tetrachloroethane	1	2	5	10	20	50	100	200
1,1,1-Trichloroethane	1	2	5	10	20	50	100	200
1,1,2,2-Tetrachloroethane	1	2	5	10	20	50	100	200
1,1,2-Trichloroethane	1	2	5	10	20	50	100	200
1,1-Dichloroethane	1	2	5	10	20	50	100	200
1,1-Dichloroethene	1	2	5	10	20	50	100	200
1,1-Dichloropropene	1	2	5	10	20	50	100	200
1,2,3-Trichlorobenzene	1	2	5	10	20	50	100	200
1,2,3-Trichloropropane	1	2	5	10	20	50	100	200
1,2,4-Trichlorobenzene	1	2	5	10	20	50	100	200
1,2,4-Trimethylbenzene	1	2	5	10	20	50	100	200
1,2-Dibromo-3-chloropropane	1	2	5	10	20	50	100	200
1,2-Dichlorobenzene	1	2	5	10	20	50	100	200
1,2-Dichloroethane	1	2	5	10	20	50	100	200
1,2-Dichloropropane	1	2	5	10	20	50	100	200
1,3,5-Trimethylbenzene	1	2	5	10	20	50	100	200
1,3-Dichlorobenzene	1	2	5	10	20	50	100	200
1,3-Dichloropropane	1	2	5	10	20	50	100	200
1,4-Dichlorobenzene	1	2	5	10	20	50	100	200
1,4-Dioxane	50	100	250	500	1,000	2,500	5,000	10,000
1-Chlorohexane	1	2	5	10	20	50	100	200
2,2-Dichloropropane	1	2	5	10	20	50	100	200
2-Butanone	4	8	20	40	80	200	400	800
2-Chloro-1,3-butadiene	1	2	5	10	20	50	100	200
2-Chlorotoluene	1	2	5	10	20	50	100	200
2-Hexanone	4	8	20	40	80	200	400	800
4-Chlorotoluene	1	2	5	10	20	50	100	200
4-Isopropyltoluene	1	2	5	10	20	50	100	200
4-Methyl-2-pentanone	4	8	20	40	80	200	400	800
Acetone	4	8	20	40	80	200	400	800
Acetonitrile	4	8	20	40	80	200	400	800
Acrolein	4	8	20	40	80	200	400	800
Acrylonitrile	4	2	5	10	20	50	100	200

Table 4. Soil Calibration Levels, 5-gram Purge¹ (Standard Mixes: MV-Main & MV-Main GasKe)

((Standard Mixes: MV-Main & MV-Main GasKe)											
			Са	libration	Level, µg/	Kg						
Compound	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7	Level 8				
Benzene	1	2	5	10	20	50	100	200				
Bromobenzene	1	2	5	10	20	50	100	200				
Bromoform	1	2	5	10	20	50	100	200				
Bromomethane	1	2	5	10	20	50	100	200				
Carbon tetrachloride	1	2	5	10	20	50	100	200				
Chlorobenzene	1	2	5	10	20	50	100	200				
Chlorobromomethane	1	2	5	10	20	50	100	200				
Chloroethane	1	2	5	10	20	50	100	200				
Chloroform	1	2	5	10	20	50	100	200				
Chloromethane	1	2	5	10	20	50	100	200				
cis-1,2-Dichloroethene	1	2	5	10	20	50	100	200				
cis-1,3-Dichloropropene	1	2	5	10	20	50	100	200				
Cyclohexanone	40	80	200	400	800	2,000	4,000	8,000				
Chlorodibromomethane	1	2	5	10	20	50	100	200				
Dibromomethane	1	2	5	10	20	50	100	200				
Dichlorobromomethane	1	2	5	10	20	50	100	200				
Dichlorodifluoromethane	1	2	5	10	20	50	100	200				
Ethanol	50	100	250	500	1,000	2,500	5,000	10,000				
Ethylbenzene	1	2	5	10	20	50	100	200				
Ethylene dibromide	1	2	5	10	20	50	100	200				
Hexachlorobutadiene	1	2	5	10	20	50	100	200				
lodomethane	1	2	5	10	20	50	100	200				
Isobutyl alcohol	20	40	100	200	400	1,000	2,000	4,000				
Isopropyl ether	5	10	25	50	100	250	500	1,000				
Isopropylbenzene	1	2	5	10	20	50	100	200				
m- and p-Xylenes	2	4	10	20	40	100	200	400				
Methacrylonitrile	10	20	50	100	200	500	1,000	2,000				
Methylene chloride	1	2	5	10	20	50	100	200				
Naphthalene	1	2	5	10	20	50	100	200				
n-Butanol	30	60	150	300	600	1,500	3,000					
n-Butylbenzene	1	2	5	10	20	50	100	200				
n-Propylbenzene	1	2	5	10	20	50	100	200				
o-Xylene	1	2	5	10	20	50	100	200				
Propionitrile	10	20	50	100	200	500	1,000	2,000				

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Table 4. Soil Calibration Levels, 5-gram Purge¹ (Standard Mixes: MV-Main & MV-Main GasKe)

			Са	libration	Level, µg/	Kg		
Compound	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7	Level 8
sec-Butylbenzene	1	2	5	10	20	50	100	200
Styrene	1	2	5	10	20	50	100	200
2-Methyl-2-propanol (tert-Butyl alcohol)	20	40	100	200	400	1,000	2,000	4,000
tert-Butylbenzene	1	2	5	10	20	50	100	200
Tetrachloroethene	1	2	5	10	20	50	100	200
Toluene	1	2	5	10	20	50	100	200
trans-1,2-Dichloroethene	1	2	5	10	20	50	100	200
trans-1,3-Dichloropropene	1	2	5	10	20	50	100	200
Trichloroethene	1	2	5	10	20	50	100	200
Trichlorofluoroethane	1	2	5	10	20	50	100	200
Vinyl chloride	1	2	5	10	20	50	100	200

¹Standards are spiked at all levels. A minimum of 5 points are used for each calibration model. Low points below the RL are routinely dropped and the high point might also be dropped for some analytes.

Table 4	A: Soil Cal	ibration L	evels, 5-g	ram Purg	e, μg/Kg¹						
(Standards: MV-Supp Std and MV-2 Cleve)											
Compound	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7				
1,1,1-Trifluoro-2,2-dichloro- ethane	2	5	10	20	50	100	200				
1,1,2-Trichloro-1,2,2- trifluoroethane	2	5	10	20	50	100	200				
1,2,3-Trimethylbenzene	2	5	10	20	50	100	200				
1,2-Dichloro-1,1,2,2- tetrafluroethane	2	5	10	20	50	100	200				
1,2-Dichloro-1,1,2- trifluoroethane	2	5	10	20	50	100	200				
2-Chloroethyl vinyl ether	2	5	10	20	50	100	200				
2-Nitropropane	2	5	10	20	50	100	200				
2-Pentanone	8	20	40	80	200	400	800				
3-Chloro-1-propene (Allyl Chloride)	2	5	10	20	50	100	200				
Carbon disulfide	2	5	10	20	50	100	200				
cis-1,4-Dichloro-2-butene	2	5	10	20	50	100	200				
Cyclohexane	2	5	10	20	50	100	200				

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Table 4A:	Soil Cal	ibration L	evels, 5-g	ram Purg	e, μg/Kg¹		
(St	andards:	MV-Supp	Std and I	MV-2 Clev	/e)		
Compound	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7
Dichlorofluoromethane	2	5	10	20	50	100	200
Ethyl Acetate	4	10	20	40	100	200	400
Ethyl ether	2	5	10	20	50	100	200
Ethyl methacrylate	4	10	20	40	100	200	400
Ethylene Oxide	250	625	1,250	2,500	6,250	12,500	25,000
Hexane	2	5	10	20	50	100	200
Isopropyl alcohol	40	100	200	400	1000	2000	4,000
Methyl acetate	10	25	50	100	250	500	1,000
Methyl methacrylate	4	10	20	40	100	200	400
Methyl tert-butyl ether (MTBE)	2	5	10	20	50	100	200
Methylcyclohexane	2	5	10	20	50	100	200
sec-Butyl alcohol	60	150	300	600	1500	3000	6,000
tert-Amyl methyl ether	10	25	50	100	250	500	1,000
tert-Butyl ethyl ether	10	25	50	100	250	500	1,000
Tetrahydrofuran	4	10	20	40	100	200	400
trans-1,4-Dichloro-2-butene	2	5	10	20	50	100	200
Trichlorofluoromethane	2	5	10	20	50	100	200
Vinyl acetate	4	10	20	40	100	200	400

¹Standards are spiked at all levels. A minimum of 5 points are used for each calibration model. Low points below the RL are routinely dropped and the high point might also be dropped for some analytes.

Table 5: Water 8260 List Calibration Levels $(\mu g/L)^1$ (Standards: MV-Main and MV-Main GasKe)

Compound	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7
1,1,1,2-Tetrachloroethane	0.3	1.0	2.0	5.0	10	30	60
1,1,1-Trichloroethane	0.3	1.0	2.0	5.0	10	30	60
1,1,2,2-Tetrachloroethane	0.3	1.0	2.0	5.0	10	30	60
1,1,2-Trichloroethane	0.3	1.0	2.0	5.0	10	30	60
1,1-Dichloroethane	0.3	1.0	2.0	5.0	10	30	60
1,1-Dichloroethene	0.3	1.0	2.0	5.0	10	30	60
1,1-Dichloropropene	0.3	1.0	2.0	5.0	10	30	60
1,2,3-Trichlorobenzene	0.3	1.0	2.0	5.0	10	30	60
1,2,3-Trichloropropane	0.3	1.0	2.0	5.0	10	30	60
1,2,4-Trichlorobenzene	0.3	1.0	2.0	5.0	10	30	60
1,2,4-Trimethylbenzene	0.3	1.0	2.0	5.0	10	30	60
1,2-Dibromo-3-chloropropane	0.3	1.0	2.0	5.0	10	30	60
1,2-Dichlorobenzene	0.3	1.0	2.0	5.0	10	30	60
1,2-Dichloroethane	0.3	1.0	2.0	5.0	10	30	60
1,2-Dichloropropane	0.3	1.0	2.0	5.0	10	30	60
1,3,5-Trimethylbenzene	0.3	1.0	2.0	5.0	10	30	60
1,3-Dichlorobenzene	0.3	1.0	2.0	5.0	10	30	60
1,3-Dichloropropane	0.3	1.0	2.0	5.0	10	30	60
1,4-Dichlorobenzene	0.3	1.0	2.0	5.0	10	30	60
1,4-Dioxane	15	50	100	250	500	1,500	3,000
1-Chlorohexane	0.3	1.0	2.0	5.0	10	30	60
2,2-Dichloropropane	0.3	1.0	2.0	5.0	10	30	60
2-Butanone (MEK)	1.2	4.0	8.0	20	40	120	240
2-Chloro-1,3-butadiene (chloroprene)	0.3	1.0	2.0	5.0	10	30	60
2-Chlorotoluene	0.3	1.0	2.0	5.0	10	30	60
2-Hexanone	1.2	4.0	8.0	20	40	120	240
2-Methyl-2-propanol (tert-Butyl alcohol)	6	20	40	100	200	600	1,200
4-Chlorotoluene	0.3	1.0	2.0	5.0	10	30	60
4-Isopropyltoluene	0.3	1.0	2.0	5.0	10	30	60
4-Methyl-2-pentanone	1.2	4.0	8.0	20	40	120	240
Acetone	1.2	4.0	8.0	20	40	120	240
Acetonitrile	3	10	20	50	100	300	600
Acrolein	3	10	20	50	100	300	600
Acrylonitrile	3	10	20	50	100	300	600

Table 5: Water 8260 List Calibration Levels $(\mu g/L)^1$ (Standards: MV-Main and MV-Main GasKe)

Compound	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7
Benzene	0.3	1.0	2.0	5.0	10	30	60
Bromobenzene	0.3	1.0	2.0	5.0	10	30	60
Bromoform	0.3	1.0	2.0	5.0	10	30	60
Bromomethane	0.3	1.0	2.0	5.0	10	30	60
Carbon tetrachloride	0.3	1.0	2.0	5.0	10	30	60
Chlorobenzene	0.3	1.0	2.0	5.0	10	30	60
Chlorobromomethane	0.3	1.0	2.0	5.0	10	30	60
Chlorodibromomethane	0.3	1.0	2.0	5.0	10	30	60
Chloroethane	0.3	1.0	2.0	5.0	10	30	60
Chloroform	0.3	1.0	2.0	5.0	10	30	60
Chloromethane	0.3	1.0	2.0	5.0	10	30	60
cis-1,2-Dichloroethene	0.3	1.0	2.0	5.0	10	30	60
cis-1,3-Dichloropropene	0.3	1.0	2.0	5.0	10	30	60
Cyclohexanone	12	40	80	200	400	1,200	2,400
Dibromomethane	0.3	1.0	2.0	5.0	10	30	60
Dichlorobromomethane	0.3	1.0	2.0	5.0	10	30	60
Dichlorodifluoromethane	0.3	1.0	2.0	5.0	10	30	60
Ethanol	15	50	100	250	500	1,500	3,000
Ethylbenzene	0.3	1.0	2.0	5.0	10	30	60
Ethylene dibromide (EDB)	0.3	1.0	2.0	5.0	10	30	60
Hexachlorobutadiene	0.3	1.0	2.0	5.0	10	30	60
lodomethane	0.3	1.0	2.0	5.0	10	30	60
Isopropyl alcohol	6	20	40	100	200	600	1,200
Isopropyl ether	1.5	5.0	10	25	50	150	300
Isopropylbenzene	0.3	1.0	2.0	5.0	10	30	60
m and p Xylenes	0.6	2.0	4.0	10	20	60	120
Methacrylonitrile	3	10	20	50	100	300	600
Methylene chloride	0.3	1.0	2.0	5.0	10	30	60
Naphthalene	0.3	1.0	2.0	5.0	10	30	60
n-Butanol	9.0	30	60	150	300	900	1,800
n-Butylbenzene	0.3	1.0	2.0	5.0	10	30	60
n-Propylbenzene	0.3	1.0	2.0	5.0	10	30	60
o-Xylene	0.3	1.0	2.0	5.0	10	30	60
Propionitrile	3.0	10	20	50	100	300	600
sec-Butylbenzene	0.3	1.0	2.0	5.0	10	30	60

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Table 5: Water 8260 List Calibration Levels (μg/L)¹ (Standards: MV-Main and MV-Main GasKe)

Compound	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7
Styrene	0.3	1.0	2.0	5.0	10	30	60
tert-Butylbenzene	0.3	1.0	2.0	5.0	10	30	60
Tetrachloroethene	0.3	1.0	2.0	5.0	10	30	60
Tetrahydrothiophene	0.3	1.0	2.0	5.0	10	30	60
Toluene	0.3	1.0	2.0	5.0	10	30	60
trans-1,2-Dichloroethene	0.3	1.0	2.0	5.0	10	30	60
trans-1,3-Dichloropropene	0.3	1.0	2.0	5.0	10	30	60
Trichloroethene	0.3	1.0	2.0	5.0	10	30	60
Trichlorofluoromethane	0.3	1.0	2.0	5.0	10	30	60
Vinyl chloride	0.3	1.0	2.0	5.0	10	30	60

¹Standards are spiked at all levels. A minimum of 5 points are used for each calibration model. Low points below the RL are routinely dropped and the high point might also be dropped for some analytes.

Table 5A: Water 8260 List Calibration Levels (μg/L)¹ (Standards: MV-Supp Std and MV-2 Cleve)

Compound	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6
1,1,1-Trifluoro-2,2-dichloroethane	1.0	2.0	5.0	10	30	60
1,1,2-Trichloro-1,2,2-trifluoroethane	1.0	2.0	5.0	10	30	60
1,2,3-Trimethylbenzene	1.0	2.0	5.0	10	30	60
1,2-Dichloro-1,1,2,2- tetrafluoroethane	1.0	2.0	5.0	10	30	60
1,2-Dichloro-1,1,2-trifluoroethane	1.0	2.0	5.0	10	30	60
2-Chloroethy vinyl ether	1.0	2.0	5.0	10	30	60
2-Nitropropane	1.0	2.0	5.0	10	30	60
2-Pentanone	4.0	8.0	20	40	120	240
3-Chloro-1-propene (Allyl chloride)	1.0	2.0	5.0	10	30	60
Carbon disulfide	1.0	2.0	5.0	10	30	60
cis-1,4-dichloro-2-butene	1.0	2.0	5.0	10	30	60
Cyclohexane	1.0	2.0	5.0	10	30	60
Dichlorofluoromethane	1.0	2.0	5.0	10	30	60
Ethyl acetate	2.0	4.0	10	20	60	120
Ethyl ether	1.0	2.0	5.0	10	30	60
Ethyl methacrylate	2.0	4.0	10	20	60	120

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Table 5A: Water 8260 List Calibration Levels (μg/L)¹ (Standards: MV-Supp Std and MV-2 Cleve)

Compound	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6
Ethylene oxide	125	250	625	1,250	3,750	7,500
Hexane	1.0	2.0	5.0	10	30	60
Isobutyl alcohol	20	40	100	200	600	1,200
Methyl acetate	5.0	10	25	50	150	300
Methylcylcohexane	1.0	2.0	5.0	10	30	60
Methyl methacrylate	2.0	4.0	8.0	20	60	120
Methyl tert-butyl ether (MTBE)	1.0	2.0	5.0	10	30	60
Propene oxide	20	100	250	500	1,500	3,000
sec-Butyl alcohol	30	60	150	300	900	1,800
tert-Amyl methyl ether	5.0	10	25	50	150	300
tert-Butyl ethyl ether	5.0	10	25	50	150	300
Tetrahydrofuran	2.0	4.0	10	20	60	120
trans-1,4-dichloro-2-butene	1.0	2.0	5.0	10	30	60
Vinyl acetate	2.0	4.0	10	20	60	120

¹Standards are spiked at all levels. A minimum of 5 points are used for each calibration model. Low points below the RL are routinely dropped and the high point might also be dropped for some analytes.

Table 6: Medium Level Soil 8260 List Calibration Levels $(\mu g/Kg)^1$ (Standards: MV-Main and MV-Main GasKe)

Compound	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7
1,1,1,2-Tetrachloroethane	0.5	1.0	2.0	5.0	10	30	60
1,1,1-Trichloroethane	0.5	1.0	2.0	5.0	10	30	60
1,1,2,2-Tetrachloroethane	0.5	1.0	2.0	5.0	10	30	60
1,1,2-Trichloroethane	0.5	1.0	2.0	5.0	10	30	60
1,1-Dichloroethane	0.5	1.0	2.0	5.0	10	30	60
1,1-Dichloroethene	0.5	1.0	2.0	5.0	10	30	60
1,1-Dichloropropene	0.5	1.0	2.0	5.0	10	30	60
1,2,3-Trichlorobenzene	0.5	1.0	2.0	5.0	10	30	60
1,2,3-Trichloropropane	0.5	1.0	2.0	5.0	10	30	60
1,2,4-Trichlorobenzene	0.5	1.0	2.0	5.0	10	30	60
1,2,4-Trimethylbenzene	0.5	1.0	2.0	5.0	10	30	60
1,2-Dibromo-3-chloropropane	0.5	1.0	2.0	5.0	10	30	60
1,2-Dichlorobenzene	0.5	1.0	2.0	5.0	10	30	60
1,2-Dichloroethane	0.5	1.0	2.0	5.0	10	30	60
1,2-Dichloropropane	0.5	1.0	2.0	5.0	10	30	60
1,3,5-Trimethylbenzene	0.5	1.0	2.0	5.0	10	30	60
1,3-Dichlorobenzene	0.5	1.0	2.0	5.0	10	30	60
1,3-Dichloropropane	0.5	1.0	2.0	5.0	10	30	60
1,4-Dichlorobenzene	0.5	1.0	2.0	5.0	10	30	60
1,4-Dioxane	25	50	100	250	500	1,500	3,000
1-Chlorohexane	0.5	1.0	2.0	5.0	10	30	60
2,2-Dichloropropane	0.5	1.0	2.0	5.0	10	30	60
2-Butanone (MEK)	2.0	4.0	8.0	20	40	120	240
2-Chloro-1,3-butadiene (chloroprene)	0.5	1.0	2.0	5.0	10	30	60
2-Chlorotoluene	0.5	1.0	2.0	5.0	10	30	60
2-Hexanone	2.0	4.0	8.0	20	40	120	240
2-Methyl-2-propanol (tert-Butyl alcohol)	10	20	40	100	200	600	1,200
4-Chlorotoluene	0.5	1.0	2.0	5.0	10	30	60
4-Isopropyltoluene	0.5	1.0	2.0	5.0	10	30	60
4-Methyl-2-pentanone	2.0	4.0	8.0	20	40	120	240
Acetone	2.0	4.0	8.0	20	40	120	240
Acetonitrile	5	10	20	50	100	300	600
Acrolein	5	10	20	50	100	300	600
Acrylonitrile	5	10	20	50	100	300	600

Table 6: Medium Level Soil 8260 List Calibration Levels $(\mu g/Kg)^1$ (Standards: MV-Main and MV-Main GasKe)

Compound	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7
Benzene	0.5	1.0	2.0	5.0	10	30	60
Bromobenzene	0.5	1.0	2.0	5.0	10	30	60
Bromoform	0.5	1.0	2.0	5.0	10	30	60
Bromomethane	0.5	1.0	2.0	5.0	10	30	60
Carbon tetrachloride	0.5	1.0	2.0	5.0	10	30	60
Chlorobenzene	0.5	1.0	2.0	5.0	10	30	60
Chlorobromomethane	0.5	1.0	2.0	5.0	10	30	60
Chlorodibromomethane	0.5	1.0	2.0	5.0	10	30	60
Chloroethane	0.5	1.0	2.0	5.0	10	30	60
Chloroform	0.5	1.0	2.0	5.0	10	30	60
Chloromethane	0.5	1.0	2.0	5.0	10	30	60
cis-1,2-Dichloroethene	0.5	1.0	2.0	5.0	10	30	60
cis-1,3-Dichloropropene	0.5	1.0	2.0	5.0	10	30	60
Cyclohexanone	20	40	80	200	400	1,200	2,400
Dibromomethane	0.5	1.0	2.0	5.0	10	30	60
Dichlorobromomethane	0.5	1.0	2.0	5.0	10	30	60
Dichlorodifluoromethane	0.5	1.0	2.0	5.0	10	30	60
Ethanol	25	50	100	250	500	1,500	3,000
Ethylbenzene	0.5	1.0	2.0	5.0	10	30	60
Ethylene dibromide (EDB)	0.5	1.0	2.0	5.0	10	30	60
Hexachlorobutadiene	0.5	1.0	2.0	5.0	10	30	60
lodomethane	0.5	1.0	2.0	5.0	10	30	60
Isopropyl alcohol	10	20	40	100	200	600	1,200
Isopropyl ether	2.5	5.0	10	25	50	150	300
Isopropylbenzene	0.5	1.0	2.0	5.0	10	30	60
m and p Xylenes	1.0	2.0	4.0	10	20	60	120
Methacrylonitrile	5	10	20	50	100	300	600
Methylene chloride	0.5	1.0	2.0	5.0	10	30	60
Naphthalene	0.5	1.0	2.0	5.0	10	30	60
n-Butanol	15	30	60	150	300	900	1,800
n-Butylbenzene	0.5	1.0	2.0	5.0	10	30	60
n-Propylbenzene	0.5	1.0	2.0	5.0	10	30	60
o-Xylene	0.5	1.0	2.0	5.0	10	30	60
Propionitrile	5.0	10	20	50	100	300	600
sec-Butylbenzene	0.5	1.0	2.0	5.0	10	30	60

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Table 6: Medium Level Soil 8260 List Calibration Levels (μg/Kg)¹ (Standards: MV-Main and MV-Main GasKe)

Compound	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7
Styrene	0.5	1.0	2.0	5.0	10	30	60
tert-Butylbenzene	0.5	1.0	2.0	5.0	10	30	60
Tetrachloroethene	0.5	1.0	2.0	5.0	10	30	60
Tetrahydrothiophene	0.5	1.0	2.0	5.0	10	30	60
Toluene	0.5	1.0	2.0	5.0	10	30	60
trans-1,2-Dichloroethene	0.5	1.0	2.0	5.0	10	30	60
trans-1,3-Dichloropropene	0.5	1.0	2.0	5.0	10	30	60
Trichloroethene	0.5	1.0	2.0	5.0	10	30	60
Trichlorofluoromethane	0.5	1.0	2.0	5.0	10	30	60
Vinyl chloride	0.5	1.0	2.0	5.0	10	30	60

¹Standards are spiked at all levels. A minimum of 5 points are used for each calibration model. Low points below the RL are routinely dropped and the high point might also be dropped for some analytes.

Table 6A: Medium Level Soil 8260 List Calibration Levels $(\mu g/Kg)^1$ (Standards: MV-Supp Std and MV-2 Cleve)

Compound	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7
1,1,1-Trifluoro-2,2-dichloroethane	0.5	1.0	2.0	5.0	10	30	60
1,1,2-Trichloro-1,2,2-trifluoroethane	0.5	1.0	2.0	5.0	10	30	60
1,2,3-Trimethylbenzene	0.5	1.0	2.0	5.0	10	30	60
1,2-Dichloro-1,1,2,2- tetrafluoroethane	0.5	1.0	2.0	5.0	10	30	60
1,2-Dichloro-1,1,2-trifluoroethane	0.5	1.0	2.0	5.0	10	30	60
2-Chloroethy vinyl ether	0.5	1.0	2.0	5.0	10	30	60
2-Nitropropane	0.5	1.0	2.0	5.0	10	30	60
2-Pentanone	2.0	4.0	8.0	20	40	120	240
3-Chloro-1-propene (Allyl chloride)	0.5	1.0	2.0	5.0	10	30	60
Carbon disulfide	0.5	1.0	2.0	5.0	10	30	60
cis-1,4-dichloro-2-butene	0.5	1.0	2.0	5.0	10	30	60
Cyclohexane	0.5	1.0	2.0	5.0	10	30	60
Dichlorofluoromethane	0.5	1.0	2.0	5.0	10	30	60
Ethyl acetate	1.0	2.0	4.0	10	20	60	120
Ethyl ether	0.5	1.0	2.0	5.0	10	30	60
Ethyl methacrylate	1.0	2.0	4.0	10	20	60	120

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Table 6A: Medium Level Soil 8260 List Calibration Levels (μg/Kg)¹ (Standards: MV-Supp Std and MV-2 Cleve)

Compound	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7
Ethylene oxide	62.5	125	250	625	1,250	3,750	7,500
Hexane	0.5	1.0	2.0	5.0	10	30	60
Isobutyl alcohol	10	20	40	100	200	600	1,200
Methyl acetate	2.5	5.0	10	25	50	150	300
Methylcylcohexane	0.5	1.0	2.0	5.0	10	30	60
Methyl methacrylate	1.0	2.0	4.0	8.0	20	60	120
Methyl tert-butyl ether (MTBE)	0.5	1.0	2.0	5.0	10	30	60
Propene oxide	10	20	100	250	500	1,500	3,000
sec-Butyl alcohol	15	30	60	150	300	900	1,800
tert-Amyl methyl ether	2.5	5.0	10	25	50	150	300
tert-Butyl ethyl ether	2.5	5.0	10	25	50	150	300
Tetrahydrofuran	1.0	2.0	4.0	10	20	60	120
trans-1,4-dichloro-2-butene	0.5	1.0	2.0	5.0	10	30	60
Vinyl acetate	1.0	2.0	4.0	10	20	60	120

¹Standards are spiked at all levels. A minimum of 5 points are used for each calibration model. Low points below the RL are routinely dropped and the high point might also be dropped for some analytes.

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Table 7. Manually Added Internal Standards

Internal Standard	Standard Concentration (µg/mL)	Quantitation Ion
Fluorobenzene	20	96
Chlorobenzene-d₅	20	119
1,4-Dichlorobenzene-d ₄	20	152

NOTES:

- 1) 10 μ L of the internal standard is added to the sample. This results in a concentration of each internal standard in the sample at 10 μ g/L for a 20 mL purge.
- 2) Except for high-level soils, the surrogate and internal standards may be combined in one solution.

Table 7A. Automatically Added Internal Standards

Internal Standard	Standard Concentration (µg/mL)	Quantitation Ion
Fluorobenzene	250	96
Chlorobenzene-d ₅	250	119
1,4-Dichlorobenzene-d ₄	250	152

NOTES:

- 1 μL of the internal standard is added to the sample. This results in a concentration of each internal standard in the sample at 10 μg/L for a 20 mL purge.
- 2) There may be some variability in the size of the internal standard loop from one instrument to the next. This is compensated for on the day of initial calibration by comparing the manually added and automatically added internal standard concentrations.

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Table 8. Manually Added Surrogate Standards

Surrogate Compounds	Standard Concentration (µg/mL)
1,2-Dichloroethane-d₄	20
Dibromofluoromethane	20
Toluene-d ₈	20
4-Bromofluorobenzene	20

NOTES:

- 1) 10 μ L of the surrogate standard is added to the sample. This results in a concentration of each surrogate in the sample at 10 μ g/L for a 20 mL purge.
- 2) Except for high-level soils, the surrogate and internal standards may be combined in one solution.
- 3) Recovery limits for surrogates are generated from historical data and are maintained by the QA department.

Table 8A. Surrogate Standards

Surrogate Compounds	Standard Concentration (µg/mL)
1,2-Dichloroethane-d₄	250
Dibromofluoromethane	250
Toluene-d ₈	250
4-Bromofluorobenzene	250

NOTES:

- 1) 1 μ L of the surrogate standard is added to the sample. This results in a concentration of each surrogate in the sample at 10 μ g/L for a 20 mL purge.
- 2) There may be some variability in the size of the surrogate standard loop from one instrument to the next. This is compensated for on the day of initial calibration by comparing the manually added and automatically added surrogate standard concentrations.
- 3) Recovery limits for surrogates are generated from historical data and are maintained by the QA department.

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Table 9. Matrix Spike and LCS Standard

Compound	Standard Concentration µg /mL
1,1-Dichloroethene	40
Methylene Chloride	40
Trans-1,2-Dichloroethene	40
1,1-Dichloroethane	40
1111-Trichloroethane	40
Carbon Tetrachloride	40
Benzene	40
Trichloroethene	40
1,2-Dichloropropane	40
Bromodichloromethane	40
Toluene	40
Tetrachloroethene	40
Chlorobenzene	40
Ethylbenzene	40
1,4-Dichlorobenzene	40
1,3-Dichlorobenzene	40

NOTES:

- 1) 2.5 μ L of the standard is added to the LCS or matrix spike sample. This results in a concentration of each spike analyte in the sample of 5 μ g/L for a 20 mL purge.
- 2) Recovery and precision limits for the LCS, MS, and MSD are generated from historical data and are maintained by the QA department.
- 3) Full analyte spikes or different compounds may also be used at the laboratory's option or at client request.

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Table 10. BFB Key Ion Abundance Criteria

Mass	Ion Abundance Criteria	
50	15 to 40 % of Mass 95	
75	30 to 60 % of Mass 95	
95	Base Peak, 100 % Relative Abundance	
96	5 to 9 % of Mass 95	
173	Less than 2 % of Mass 174	
174	Greater than 50 % of Mass 95	
175	5 to 9 % of Mass 174	
176	Greater than 95 %, but less than 101 % of Mass 174	
177	5 to 9 % of Mass 176	

Table 11. SPCC Compounds and Minimum Response Factors

Compound	8260B Min. RF
Chloromethane	0.100
1,1-Dichloroethane	0.100
Bromoform	> 0.100
1,1,2,2-Tetrachloroethane	0.300
Chlorobenzene	0.300

Table 12. CCC Compounds

Compound	Max. %RSD from Initial Calibration	Max. %D for continuing calibration
Vinyl Chloride	≤ 30.0	≤ 20.0
1,1-Dichloroethene	≤ 30.0	≤ 20.0
Chloroform	≤ 30.0	≤ 20.0
1,2-Dichloropropane	≤ 30.0	≤ 20.0
Toluene	≤ 30.0	≤ 20.0
Ethylbenzene	≤ 30.0	≤ 20.0

Table 13. Characteristic lons

Compound	Primary*	Secondary	Tertiary
1,2-Dichloroethane-d ₄ (Surrogate)	65	102	
Dichlorodifluoromethane	85	87	50, 101,103
Dibromofluoromethane	111	113	
Chloromethane	50	52	49
Vinyl chloride	62	64	61
Bromomethane	94	96	79
Chloroethane	64	66	49
Trichlorofluoromethane	101	103	66
1,1-Dichloroethene	96	61	98
Acrolein	56	55	58
Iodomethane	142	127	141
Carbon disulfide	76	78	
Trichlorotrifluoroethane	151	101	153
Ethanol	45	46	
Acetone	43	58	
Methylene chloride	84	49	51, 86
Tert-Butyl alcohol	59	74	
Trans-1,2-Dichloroethene	96	61	98
Acrylonitrile	53	52	51
Methyl tert butyl ether	73		
Hexane	57	43	
1,1-Dichloroethane	63	65	83
cis-1,2-Dichloroethene	96	61	98
2-Butanone	43	72**	
Tetrahydrofuran	42	71	
Chloroform	83	85	47
1,2-Dichloroethane	62	64	98
Dibromomethane	93	174	95, 172, 176
1,4-Dioxane	88	58	
Vinyl acetate	43	86	
1,1,1-Trichloroethane	97	99	117
Carbon tetrachloride	117	119	121

Table 13. Characteristic lons (cont.)

Compound	Primary*	Secondary	Tertiary
Benzene	78	52	77
Trichloroethene	95	130***	97, 132
1,2-Dichloropropane	63	65	41
Bromodichloromethane	83	85	129
2-Chloroethyl vinyl ether	63	65	106
cis-1,3-Dichloropropene	75	77	39
trans-1,3-Dichloropropene	75	77	39
1,1,2-Trichloroethane	97	83	85, 99
Chlorodibromomethane	129	127	131
Bromoform	173	171	175, 252
1,2,3-Trichloropropane	75	110	77, 112, 97
Toluene-d ₈ (Surrogate)	98	70	100
4-Bromofluorobenzene (Surrogate)	95	174	176
Toluene	91	92	65
4-Methyl-2-pentanone	43	58	57, 100
Tetrachloroethene	164	166	131
Ethyl methacrylate	69	41	99, 86, 114
2-Hexanone	43	58	57, 100
Chlorobenzene	112	114	77
Ethylbenzene	106	91	
Xylenes	106	91	
Styrene	104	103	78, 51, 77
Dichlorobenzene (all isomers)	146	148	111
Trans 1,4-Dichloro-2-butene	53	75	89, 77, 124
1,1,2,2-Tetrachloroethane	83	85	131, 133
Allyl Chloride	41	76	78
Acetonitrile	41	40	
Dichlorofluoromethane	67	69	
Isopropyl ether	87	59	45
Chloroprene	53	88	90
n-Butanol	56	41	42
Propionitrile	54	52	55
Methacrylonitrile	41	67	52
Isobutanol	41	43	74

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Table 13. Characteristic lons (cont.)

Compound	Primary*	Secondary	Tertiary
Methyl methacrylate	41	69	100
1,1,1,2-Tetrachloroethane	131	133	119
1,2-Dibromo-3-chloropropane	157	155	75
Ethyl ether	59	74	
Ethyl Acetate	43	88	61
2-Nitropropane	41	43	46
Cyclohexanone	55	42	98
Isopropylbenzene	105	120	
2,2-Dichloropropane	77	97	
Bromochloromethane	128	49	130
1,1-Dichloropropene	75	39	110
1,3-Dichloropropane	76	41	78
1-Chlorohexane	91	55	41
1,1,1,2-Tetrachloroethane	131	133	
Bromobenzene	156	158	77
n-Propylbenzene	120	91	65
2-Chlorotoluene	126	91	65
1,3,5-Trimethylbenzene	105	120	77
4-Chlorobenzene	126	91	89
t-Butylbenzene	119	134	91
sec-Butylbenzene	134	105	
4-Isopropyltoluene	119	134	91
n-Butylbenzene	91	92	134
1,2,4-Trichlorobenzene	180	182	
Hexachlorobutadiene	225	227	223
Naphthalene	128	127	
1,2,3-Trichlorobenzene	180	182	

^{*} The primary ion should be used for quantitation unless interferences are present, in which case a secondary ion may be used.

^{**} m/z 43 may be used for quantitation of 2-butanone, but m/z 72 <u>must</u> be present for positive identification.

^{***} Used as quantitation ion for method 624.

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Table 14. State of Arizona ICV/CCV Quality Control Limits

QC Limits not specified in method	Default QC (method specified or laboratory historical if not specified)
CCV Non-CCC compounds	CCC limits (≤30%)
ICV	Same as CCV (≤30%)
Reporting Limit	Must be supported by low level initial calibration standard
LCS/LCSD	Lab historical
MS/MSD	Lab historical

NOTES:

1) Based on ADHS Rule A.A.C.R9-14-615.C.8. Director approved on June 29, 2005 for the labs to use default limits as an alternative to developing statistically derived limits.

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Table 15. List 1 Poorly Performing Compounds

The laboratory's GC/MS group identified this list of compounds based on current and historical performance. The recovery performance was reviewed against full spike recovery data and method performance data, where available, to validate each compound as a "poor performer."

Acetone	1,2-Dichloro-1,1,2,2-tetrafluoroethane
Acetonitrile	Ethanol
Acrolein	Ethyl acetate
Acrylonitrile	Ethylene oxide
n-Butanol	2-Hexanone
2-Butanone (MEK)	Isobutyl alcohol
tert-Butyl alcohol	Isopropanol
Carbon disulfide	Methacrylonitrile
2-Chloroethyl vinyl ether	Methyl acetate
2-Chloro-1,1,1-trifluoroethane	4-Methyl-2-pentanone
Chlorotrifluoroethene	2-Nitropropane
cis-1,4-Dichloro-2-butene	2-Pentanone
trans-1,4-Dichloro-2-butene	2-Propanol
Dichlorodifluoromethane	Propionitrile
Dichlorofluoromethane	Tetrahydrofuran
1,2-Dibromo-3-chloropropane (DBCP)	Tetrahydrothiophene
1,2-Dichlorotetrafluoroethane	1,1,2-Trichloro-1,2,2-trifluoroethane
1,2-Dichloro-1,1,2-trifluoroethane (Freon 123a)	Trichlorofluoromethane
2,2-Dichloro-1,1,1-trifluoroethane	Vinyl acetate
1,4-Dioxane	

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Table A-1. Method 624 Analytes and Reporting Limits, 5-mL Purge

Analytes	μg/L
Acrolein ¹	100
Acrylonitrile ¹	100
Benzene	5
Bromodichloromethane	5
Bromoform	5
Bromomethane	10
Carbon tetrachloride	5
Chlorobenzene	5
Chloroethane	10
2-Chloroethyl vinyl ether	5
Chloroform	5
Chloromethane	10
Dibromochloromethane	5
1,2-Dichlorobenzene	5
1,3-Dichlorobenzene	5
1,4-Dichlorobenzene	5
1,1-Dichloroethane	5
1,2-Dichloroethane	5
1,1-Dichloroethene	5
trans-1,2-Dichloroethene	5
1,2-Dichloropropane	5
cis-1,3-Dichloropropene	10
trans-1,3-Dichloropropene	5
Ethylbenzene	5
Methylene chloride	5
1,1,2,2-Tetrachloroethane	5
Tetrachloroethene	5
Toluene	5
1,1,1-Trichloroethane	5
1,1,2-Trichloroethane	5
Trichloroethene	5
Trichlorofluoromethane	15
Vinyl chloride	10

Acrolein and Acrylonitrile have been added to the 624 analyte list in the EPA Method Update Rule, May 18, 2012. Analysis of these analytes by Method 624 as being regulatory compliant is dependent upon individual state approval of the MUR. Verify state status before analysis.

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Table A-2. Method 624 QC Acceptance Criteria

Analytes ¹	Daily QC Check (CCV) Acceptance Criteria (20 μg/L spike)	Mean Recovery, Initial Demonstration Acceptance Criteria (IDOC) (20 µg/L spike)	Std Dev, Initial Demonstration Acceptance Criteria (IDOC) (20 µg/L spike)	Matrix Spike and LCS Acceptance Criteria (% Recovery)
Acrolein ²	45.9-54.1	42.9-60.1	4.6	88-118
Acrylonitrile ²	41.2-58.8	33.1-66.9	9.9	71-135
Benzene	12.8 - 27.2	15.2 - 26.0	6.9	37 - 151
Bromodichloromethane	13.1 - 26.9	10.1 - 28.0	6.4	35 - 155
Bromoform	14.2 - 25.8	11.4 - 31.1	5.4	45 - 169
Bromomethane	2.8 - 37.2	D - 41.2	17.9	D - 242
Carbon tetrachloride	14.6 - 25.4	17.2 - 23.5	5.2	70 - 140
Chlorobenzene	13.2 - 26.8	16.4 - 27.4	6.3	37 - 160
Chloroethane	7.6 - 32.4	8.4 - 40.4	11.4	14 - 230
2-Chloroethyl vinyl ether	D - 44.8	D - 50.4	25.9	D - 305
Chloroform	13.5 - 26.5	13.7 - 24.2	6.1	51 - 138
Chloromethane	D - 40.8	D - 45.9	19.8	D - 273
Dibromochloromethane	13.5 - 26.5	13.8 - 26.6	6.1	53 - 149
1,2-Dichlorobenzene	12.6 - 27.4	11.8 - 34.7	7.1	18 - 190
1,3-Dichlorobenzene	14.6 - 25.4	17.0 - 28.8	5.5	59 - 156
1,4-Dichlorobenzene	12.6 - 27.4	11.8 - 34.7	7.1	18 - 190
1,1-Dichloroethane	14.5 - 25.5	14.2 - 28.5	5.1	59 - 155
1,2-Dichloroethane	13.6 - 26.4	14.3 - 27.4	6.0	49 - 155
1,1-Dichloroethene	10.1 - 29.9	3.7 - 42.3	9.1	D - 234
trans-1,2-Dichloroethene	13.9 - 26.1	13.6 - 28.5	5.7	54 - 156
1,2-Dichloropropane	6.8 - 33.2	3.8 - 36.2	13.8	D - 210
cis-1,3-Dichloropropene	4.8 - 35.2	1.0 - 39.0	15.8	D- 227
trans-1,3-Dichloropropene	10.0 - 30.0	7.6 - 32.4	10.4	17- 183
Ethylbenzene	11.8 - 28.2	17.4 - 26.7	7.5	37 - 162
Methylene chloride	12.1 - 27.9	D - 41.0	7.4	D - 221
1,1,2,2-Tetrachloroethane	12.1 - 27.9	13.5 - 27.2	7.4	46 - 157
Tetrachloroethene	14.7 - 25.3	17.0 - 26.6	5.0	64 - 148
Toluene	14.9 - 25.1	16.6 - 26.7	4.8	47 - 150
1,1,1-Trichloroethane	15.0 - 25.0	13.7 - 30.1	4.6	52 - 162
1,1,2-Trichloroethane	14.2 - 25.8	14.3 - 27.1	5.5	52 - 150
Trichloroethene	13.3 - 26.7	18.6 - 27.6	6.6	71 - 157
Trichlorofluoromethane	9.6 – 30.4	8.9- 31.5	10.0	17 - 181
Vinyl chloride	0.8 - 39.2	D - 43.5	20.0	D - 251

Analytes not listed on the table must meet a CCV drift criteria of ± 30%. Method 624 does not specify second source (ICV) criteria. The laboratory has adopted criteria of ± 30% difference for the ICV. The LIMS requires a minimum value of 10% for the lower limit when D is noted in the reference table.

Acrolein and Acrylonitrile have been added to the 624 analyte list in the EPA Method Update Rule, May 18, 2012. Analysis of these analytes by Method 624 as being regulatory compliant is dependent upon individual state approval of the MUR. Verify state status before analysis. Per the MUR, QC criteria from Method 603 are to be applied and are presented here.

Table A-3. Calibration Levels for 624, 5 mL Purge

Compound	Level 1	Level 2	Level 3	Level 4	Level 5
Acetone	20	40	80	200	400
Acrolein	50	100	200	500	1000
Acrylonitrile	50	100	200	500	1000
Benzene	5.0	10	20	50	100
Bromoform	5.0	10	20	50	100
Bromomethane	5.0	10	20	50	100
2-Butanone	20	40	80	200	400
Carbon disulfide	5.0	10	20	50	100
Carbon tetrachloride	5.0	10	20	50	100
Chlorobenzene	5.0	10	20	50	100
Chlorodibromomethane	5.0	10	20	50	100
Chloroethane	5.0	10	20	50	100
2-Chloroethyl vinyl ether	5.0	10	20	50	100
Chloroform	5.0	10	20	50	100
Chloromethane	5.0	10	20	50	100
1,2-Dibromo-3-chloropropane	5.0	10	20	50	100
1,2-Dibromoethane (EDB)	5.0	10	20	50	100
Dibromomethane	5.0	10	20	50	100
1,2-Dichlorobenzene	5.0	10	20	50	100
1,3-Dichlorobenzene	5.0	10	20	50	100
1,4-Dichlorobenzene	5.0	10	20	50	100
Dichlorobromomethane	5.0	10	20	50	100
Dichlorodifluoromethane	5.0	10	20	50	100
1,1-Dichloroethane	5.0	10	20	50	100
1,2-Dichloroethane	5.0	10	20	50	100
cis-1,2-Dichloroethene	5.0	10	20	50	100
trans-1,2-Dichloroethene	5.0	10	20	50	100
1,1-Dichloroethene	5.0	10	20	50	100
1,2-Dichloropropane	5.0	10	20	50	100
cis-1,3-Dichloropropene	5.0	10	20	50	100
trans-1,3-Dichloropropene	5.0	10	20	50	100
1,4-Dioxane	250	500	1000	2500	5000

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Table A-3. Calibration Levels for 624, 5 mL Purge (cont.)

Compound	Level 1	Level 2	Level 3	Level 4	Level 5
Ethylbenzene	5.0	10	20	50	100
Hexane	5.0	10	20	50	100
2-Hexanone	20	40	80	200	400
Methylene chloride	5.0	10	20	50	100
4-Methyl-2-pentanone (MIBK)	20	40	80	200	400
Methyl tert-butyl ether (MTBE)	5.0	10	20	50	100
Styrene	5.0	10	20	50	100
1,1,1,2-Tetrachloroethane	5.0	10	20	50	100
1,1,2,2-Tetrachloroethane	5.0	10	20	50	100
Tetrachloroethene	5.0	10	20	50	100
Toluene	5.0	10	20	50	100
1,1,1-Trichloroethane	5.0	10	20	50	100
1,1,2-Trichloroethane	5.0	10	20	50	100
Trichloroethene	5.0	10	20	50	100
Trichlorofluoromethane	5.0	10	20	50	100
1,2,3-Trichloropropane	5.0	10	20	50	100
Vinyl acetate	5.0	10	20	50	100
Vinyl chloride	5.0	10	20	50	100
m- and p-Xylenes	10	20	40	100	200
o-Xylene	5.0	10	20	50	100

If the response factor (RF) is constant over the working range (<35% RSD), the average RF may used for calculations. Alternatively, a calibration curve may be used if the correlation coefficient is ≥ 0.99 .

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APPENDIX A

Modifications for Analysis of 1,4-Dioxane, 1,2,3-Trichloropropane, 1,2-Dibromo-3chloropropane, and 1,2-Dibromoethane by Selected Ion Monitoring

1.0 REQUIREMENTS FOR METHOD 8260 SELECTED ION MONITORING (SIM)

- 1.1 The gas chromatograph/mass spectrometer (GCMS) is utilized in the SIM mode to obtain lower reporting limits. The standard analyte list and reporting limits are listed in Table Ap-1.
- 1.2 This method can be applied to aqueous and solid matrices.
- 1.3 The sample preparation is the same as defined in section 10.1.1 through 10.1.3 in this SOP, DV-MS-0010.
- 1.4 The tune period for this method is defined as 12 hours. Instrument tuning is described in section 10.1.11.3 above.
- 1.5 Initial calibration curve requirements are as follows:
 - 1.5.1 Same as for 8260 detailed in Section 10.1.12 of this SOP.
 - 1.5.2 The calibrations levels are shown in Table Ap-2.
- 1.6 Continuing calibration verification requirements are as follows:
 - 1.6.1 The %drift for 1,4-dioxane must be \leq 25% for the continuing calibration to be valid.
 - 1.6.2 In addition, the %drift for the surrogate compounds should be $\leq 25\%$.
- 1.7 Matrix Spike and LCS requirements are as follows:
 - 1.7.1 The spike levels are listed in Table Ap-3.
- 1.8 Internal Standards: The internal standard concentrations are listed in Table Ap-5.
- 1.9 Surrogates: The surrogate concentrations are listed in Table Ap-4.
- 1.10 Instrument Conditions are shown in Table Ap-7.

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Table Ap-1.

TAL Method 8260SIM Standard Reporting Limits

Analytes	CAS Number	Aqueous, μg/L	Solid, μg/Kg
1,4-Dioxane	123-91-1	2.0	5.0
1,2-Dibromo-3- chloropropane	96-12-8	0.02	1.0
1,2-Dibromoethane	106-93-4	0.02	1.0
1,2,3-Trichloropropane	96-18-4	0.02	1.0

Table Ap-2.

Method 8260SIM Calibration Levels

Calibration Level	1,4-Dioxane Aqueous Calibration Concentration, μg/L	EDB,DBCP,TCP Aqueous Calibration Concentration, μg/L	1,4-Dioxane Solid Calibration Concentration, μg/Kg	EDB,DBCP,TCP Solid Calibration Concentration, µg/Kg
1	NA	0.02	1.0	1.0
2	NA	0.05	2.0	2.0
3	0.2	0.2	4.0	4.0
4	1.0	1.0	8.0	8.0
5	2.0	2.0	16.0	16.0
6	5.0	5.0	32.0	32.0
7	10.0	10.0	48.0	48.0
8	20.0	20.0	NA	NA
SSV	5.0	5.0	16.0	16.0

Table Ap-3.

Method 8260SIM LCS Spike Concentrations

LCS Compounds	Aqueous Spiking Level, μg/L	Solid Spiking Level, μg/Kg
1,4-Dioxane	5.0	20
1,2-Dibromo-3-chloropropane	1.0	8
1,2-Dibromoethane	1.0	8
1,2,3-Trichloropropane	1.0	8

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Table Ap-4.

8260SIM Surrogate Compounds

Surrogate Compounds	Aqueous Spiking Level, μg/L ¹	Solid Spiking Level, μg/Kg ¹
Dibromofluoromethane	12.5	50
1,2-Dichloroethane-d₄	12.5	50
Toluene-d ₈	12.5	50
4-Bromofluorobenzene	12.5	50

^{1 —} Exact spike levels are dependent upon the calibration of the autosampler loop used for the addition of the surrogate spike solution.

Table Ap-5.

8260SIM Internal Standard Compounds

Surrogate Compounds	Aqueous Spiking Level, μg/L	Solid Spiking Level, μg/Kg
Fluorobenzene	12.5	50
Chlorobenzene-d ₅	12.5	50
1,4-Dichlorobenzene-d ₄	12.5	50

Table Ap-6.

8260 Selected Masses

Compound	Quant	Qualifier Ion
1,4-Dioxane	88	58
Fluorobenzene	96	70
Chlorobenzene-d ₅	119	117
1,4-Dichlorobenzene-d ₄	152	150
Dibromofluoromethane	111	113
1,2-Dichloroethane-d ₄	65	102
Toluene-d ₈	98	70
4-Bromofluorobenzene	95	174
1,2-Dibromo-3-chloropropane	157	155
1,2-Dibromoethane	107	109
1,2,3-Trichloropropane	110	75

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Table Ap-7. **Suggested Instrument Conditions for 8260SIM**

Selected Masses:	See Table Ap-6
Dwell Time:	≥ 30 milliseconds
Initial Column Temperature/Hold Time:	50 °C for 2 minutes
Column Temperature Program:	50 - 160 °C at 30°C/min, 160 - 220 °C at 60°C/min .
Final Column Temperature/Hold Time:	220 °C/4.3 min hold
Injector Temperature:	220 °C
Transfer Line Temperature:	260 °C
Source Temperature:	240 °C
Trap Desorb Temperature:	270 °C
Sample Volume:	0.5 μΙ
Carrier Gas:	Helium at 1.3mL/min.
Column:	DB-624 Capillary 60m x 0.25mm x 1.8 um film thickness, or equivalent

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APPENDIX B

Modifications for Analysis of Soils Collected for the State of Alaska

1. Collection and Preservation Requirements

Preservation and Holding Time for Volatiles in Soil Method 5035A for Alaska

Container/Contents ¹	Preservation	Holding time	Analysis
Vial containing methanol and TFT surrogate	Sample is extruded into pre-tared 4 oz jar, containing 25 mL of methanol spiked with 2.5 ppm -trifluorotoluene, cooled to $\leq 6^{\circ}$ C and frozen upon receipt at laboratory.	14 days	Medium Level

Sample weights are calculated in the laboratory by adding the received weight of the sample into the AK Methanol Volume Correction spreadsheet stored on G:\QA\Edit\FORMS\GCMS.

2. Sample Preparation for Medium-Level Analysis - Field Preserved, AK method

- a. Fill a 40 mL VOA vial with reagent water ~ 42 mL (no head space), and remove 1000 μL of water using a volumetric pipette or syringe.
- b. Add 1050 μ L of methanol extract to the vial and immediately cap. Invert the vial to ensure that there is no air bubble larger than 4 mm present. If a > 4 mm air bubble is present, re-prepare the sample.
- c. Load the sample in the auto sampler and proceed to analyze against the methanol calibration curve.
- d. As with water samples, surrogate and internal standard solutions are added by the autosampler (see Tables 7 and 7A in the main body of this SOP). The surrogate -trifluorotoluene is added to the samples at the time of sampling. Recoveries for this surrogate will be reported in addition to recoveries for the surrogate compounds added at the time of analysis.
- e. Prepare laboratory control samples by filling a 40 mL VOA vial with reagent water, and remove 1000 μ L of water using a volumetric pipette or syringe. Add reagents as needed plus sufficient methanol for a total methanol volume of 1050 μ L. The recommended concentration for the LCS is the same as the Level 5 of the initial calibration curve.
- f. Remove a portion of the methanol extract for each sample and store in a clean Teflon-capped vial with no headspace at \leq 6 °C until analysis. Duplicate aliquots of the methanol extract should be taken and stored.

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3. Percent Moisture Correction for Soils from the State of Alaska

A percent moisture correction is required for soil samples submitted from the state of AK to adjust the extraction final volume in order to allow for the miscible solvent effects. The following formula is used to determine the corrected final volume. This calculation is performed in the AK Methanol Volume Correction spreadsheet stored on G:\QA\Edit\FORMS\GCMS.

a. $V_t = [V_m + (M * W_s/100)]$

Where:

V_t = final extract volume, corrected for moisture (mL)

 V_m = volume methanol used for extraction (mL)

M = moisture content of the sample (%)

 W_s = aliquot of sample extracted (g)

Table Bp-1. TestAmerica 8260 Reporting Limits – AK Soils

Compound	CAS Number	Medium Soil μg/Kg
Dichlorodifluoromethane	75-71-8	80
Chloromethane	74-87-3	40
Bromomethane	74-83-9	40
Vinyl chloride	75-01-4	40
Chloroethane	75-00-3	40
n-Butanol	71-36-3	800
Trichlorofluoromethane	75-69-4	40
Acrolein	107-02-8	200
Acetone	67-64-1	400
Trichlorotrifluoroethane	76-13-1	400
Iodomethane	74-88-4	500
Carbon disulfide	75-15-0	40
Methylene chloride	75-09-2	40
tert-Butyl alcohol	75-65-0	800
1,1-Dichloroethene	75-35-4	40
1,1-Dichloroethane	75-34-3	40
trans-1,2-Dichloroethene	156-60-5	40
Acrylonitrile	107-13-1	400
Methyl tert-butyl ether (MTBE)	1634-04-4	200
Hexane	110-54-3	400
cis-1,2-Dichloroethene	156-59-2	40
1,2-Dichloroethene (Total)	540-59-0	40
Tetrahydrofuran	109-99-9	80
Chloroform	67-66-3	40
1,2-Dichloroethane	107-06-2	40
Dibromomethane	74-95-3	40
2-Butanone	78-93-3	160
1,4-Dioxane	123-91-1	2,000
1,1,1-Trichloroethane	71-55-6	40
Carbon tetrachloride	56-23-5	40
Bromodichloromethane	75-27-4	40
1,2-Dichloropropane	78-87-5	40
Isopropyl Alcohol	67-63-0	1,000
Isopropyl ether	108-20-3	200

Table Bp-1. TestAmerica 8260 Reporting Limits - AK Soils

Compound	CAS Number	Medium Soil μg/Kg
cis-1,3-Dichloropropene	10061-01-5	40
Trichloroethene	79-01-6	40
Dibromochloromethane	124-48-1	40
1,2-Dibromoethane	106-93-4	40
1,2,3-Trichloropropane	96-18-4	40
1,1,2-Trichloroethane	79-00-5	40
Benzene	71-43-2	16
Ethylmethacrylate	97-63-2	80
trans-1,3-Dichloropropene	10061-02-6	40
Bromoform	75-25-2	40
4-Methyl-2-pentanone	108-10-1	160
2-Hexanone	591-78-6	160
Tetrachloroethene	127-18-4	40
Toluene	108-88-3	40
1,1,2,2-Tetrachloroethane	79-34-5	40
2-Chloroethyl vinyl ether	110-75-8	80
Vinyl acetate	108-05-4	80
Chlorobenzene	108-90-7	40
Ethylbenzene	100-41-4	40
Styrene	100-42-5	40
trans-1,4-Dichloro-2-butene	110-57-6	400
m- and p-Xylenes	179601-23-1	80
o-Xylene	95-47-6	40
Total xylenes	1330-20-7	80
1,3-Dichlorobenzene	541-73-1	40
1,4-Dichlorobenzene	106-46-7	40
1,2-Dichlorobenzene	95-50-1	40
2,2-Dichloropropane	590-20-7	40
Bromochloromethane	74-97-5	40
1,1-Dichloropropene	563-58-6	40
1,3-Dichloropropane	142-28-9	40
1-Chlorohexane	544-10-5	80
1,1,1,2-Tetrachloroethane	630-20-6	40

Table Bp-1. TestAmerica 8260 Reporting Limits - AK Soils

Compound	CAS Number	Medium Soil μg/Kg
Isopropylbenzene	98-82-8	40
Bromobenzene	108-86-1	40
n-Propylbenzene	103-65-1	40
2-Chlorotoluene	95-49-8	40
4-Chlorotoluene	106-43-4	40
1,3,5-Trimethylbenzene	108-67-8	40
tert-Butylbenzene	98-06-6	40
1,2,4-Trimethylbenzene	95-63-6	40
sec-Butylbenzene	135-98-8	40
4-Isopropyltoluene	99-87-6	40
n-Butylbenzene	104-51-8	40
1,2-Dibromo-3-chloropropane	96-12-8	200
1,2,4-Trichlorobenzene	120-82-1	40
Naphthalene	91-20-3	40
Hexachlorobutadiene	87-68-3	40
1,2,3-Trichlorobenzene	87-61-6	40
Propionitrile	107-12-0	400
Cylcohexanone	108-94-1	1,600
Methyl methacrylate	80-62-6	80
Acetonitrile	75-05-8	400
Methacrylonitrile	126-98-7	400
1,2-Dichloro-1,1,2,2- Tetrafluoroethane	76-14-2	160
1,2-Dichloro-1,1,2-trifluoroethane	354-23-4	160
2-Pentanone	107-87-9	600
cis-1,4-Dichloro-2-butene	1476-11-5	400
Cyclohexane	110-82-7	40
Methyl acetate	79-20-9	200
Methylcyclohexane	108-87-2	160
2-Chloro-1,3-butadiene	126-99-8	80
2-Methyl-2-propanol	75-65-0	800

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Table Bp-1. TestAmerica 8260 Reporting Limits - AK Soils

Compound	CAS Number	Medium Soil μg/Kg
tert-Butyl ethyl ether	637-92-3	80
1,2,3-Trimethylbenzene	526-73-8	40
Ethyl acetate	141-78-6	80
Ethyl ether	60-29-7	200
Isobutyl alcohol	78-83-1	800
Dichlorofluoromethane	75-43-4	120
Tetrahydrothiophene	110-01-0	40

Reporting limits listed for soil/sediment are based on wet weight. The reporting limits calculated by the laboratory for soil/sediment, calculated on dry weight basis, will be higher.

	Table Bp-2										
Ca	Calibration Levels for 8260, 5035FM_AK (ug/Kg)										
Compound	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7	Level 8			
1,1,1,2-Tetrachloroethane	20	40	80	200	600	2000	4000	8000			
1,1,1-Trichloroethane	20	40	80	200	600	2000	4000	8000			
1,1,2,2-Tetrachloroethane	20	40	80	200	600	2000	4000	8000			
1,1,2-Trichloroethane	20	40	80	200	600	2000	4000	8000			
1,1-Dichloroethane	20	40	80	200	600	2000	4000	8000			
1,1-Dichloroethene	20	40	80	200	600	2000	4000	8000			
1,1-Dichloropropene	20	40	80	200	600	2000	4000	8000			
1,2,3-Trichlorobenzene	20	40	80	200	600	2000	4000	8000			
1,2,3-Trichloropropane	20	40	80	200	600	2000	4000	8000			
1,2,4-Trichlorobenzene	20	40	80	200	600	2000	4000	8000			
1,2,4-Trimethylbenzene	20	40	80	200	600	2000	4000	8000			
1,2-Dibromo-3-chloropropane	20	40	80	200	600	2000	4000	8000			
1,2-Dichlorobenzene	20	40	80	200	600	2000	4000	8000			
1,2-Dichloroethane	20	40	80	200	600	2000	4000	8000			
1,2-Dichloropropane	20	40	80	200	600	2000	4000	8000			
1,3,5-Trimethylbenzene	20	40	80	200	600	2000	4000	8000			
1,3-Dichlorobenzene	20	40	80	200	600	2000	4000	8000			
1,3-Dichloropropane	20	40	80	200	600	2000	4000	8000			
1,4-Dichlorobenzene	20	40	80	200	600	2000	4000	8000			
1,4-Dioxane	1000	2000	4000	10000	30000	100000	200000	400000			
1-Chlorohexane	20	40	80	200	600	2000	4000	8000			
2,2-Dichloropropane	20	40	80	200	600	2000	4000	8000			
2-Butanone (MEK)	80	160	320	800	2400	8000	16000	32000			
2-Chloro-1,3-butadiene (chloroprene)	20	40	80	200	600	2000	4000	8000			
2-Chlorotoluene	20	40	80	200	600	2000	4000	8000			
2-Hexanone	80	160	320	800	2400	8000	16000	32000			
2-Methyl-2-propanol (tert-Butyl alcohol)	400	800	1600	4000	12000	40000	80000	160000			
4-Chlorotoluene	20	40	80	200	600	2000	4000	8000			
4-Isopropyltoluene	20	40	80	200	600	2000	4000	8000			
4-Methyl-2-pentanone	80	160	320	800	2400	8000	16000	32000			
Acetone	80	160	320	800	2400	8000	16000	32000			
Acetonitrile	200	400	800	2000	6000	20000	40000	80000			
Acrolein	200	400	800	2000	6000	20000	40000	80000			
Acrylonitrile	200	400	800	2000	6000	20000	40000	80000			

	Table Bp-2									
Calibration Levels for 8260, 5035FM_AK (ug/Kg)										
Compound	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7	Level 8		
Benzene	20	40	80	200	600	2000	4000	8000		
Bromobenzene	20	40	80	200	600	2000	4000	8000		
Bromoform	20	40	80	200	600	2000	4000	8000		
Bromomethane	20	40	80	200	600	2000	4000	8000		
Carbon tetrachloride	20	40	80	200	600	2000	4000	8000		
Chlorobenzene	20	40	80	200	600	2000	4000	8000		
Chlorobromomethane	20	40	80	200	600	2000	4000	8000		
Chlorodibromomethane	20	40	80	200	600	2000	4000	8000		
Chloroethane	20	40	80	200	600	2000	4000	8000		
Chloroform	20	40	80	200	600	2000	4000	8000		
Chloromethane	20	40	80	200	600	2000	4000	8000		
cis-1,2-Dichloroethene	20	40	80	200	600	2000	4000	8000		
cis-1,3-Dichloropropene	20	40	80	200	600	2000	4000	8000		
Cyclohexanone	20	40	80	200	300	1000	2000	4000		
Dibromomethane	20	40	80	200	600	2000	4000	8000		
Dichlorobromomethane	20	40	80	200	600	2000	4000	8000		
Dichlorodifluoromethane	20	40	80	200	600	2000	4000	8000		
Ethanol	1000	2000	4000	10000	30000	100000	200000	400000		
Ethylbenzene	20	40	80	200	600	2000	4000	8000		
Ethylene dibromide (EDB)	20	40	80	200	600	2000	4000	8000		
Hexachlorobutadiene	20	40	80	200	600	2000	4000	8000		
lodomethane	20	40	80	200	600	2000	4000	8000		
Isopropyl alcohol	400	800	1600	4000	12000	40000	80000	160000		
Isopropyl ether	100	200	400	1000	3000	10000	20000	40000		
Isopropylbenzene	20	40	80	200	600	2000	4000	8000		
m- and p-Xylenes	40	80	160	400	1200	4000	8000	16000		
Methacrylonitrile	200	400	800	2000	6000	20000	40000	80000		
Methylene chloride	20	40	80	200	600	2000	4000	8000		
Naphthalene	20	40	80	200	600	2000	4000	8000		
n-Butanol	600	1200	2400	6000	18000	60000	120000	240000		
n-Butylbenzene	20	40	80	200	600	2000	4000	8000		
n-Propylbenzene	20	40	80	200	600	2000	4000	8000		
o-Xylene	20	40	80	200	600	2000	4000	8000		
Propionitrile	200	400	800	2000	6000	20000	40000	80000		
sec-Butylbenzene	20	40	80	200	600	2000	4000	8000		
Styrene	20	40	80	200	600	2000	4000	8000		

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Table Bp-2										
Calibration Levels for 8260, 5035FM_AK (ug/Kg)										
Compound	Compound Level 1 Level 2 Level 3 Level 4 Level 5 Level 6 Level 7 Level 8									
tert-Butylbenzene	20	40	80	200	600	2000	4000	8000		
Tetrachloroethene	20	40	80	200	600	2000	4000	8000		
Tetrahydrothiophene	20	40	80	200	600	2000	4000	8000		
Toluene	20	40	80	200	600	2000	4000	8000		
trans-1,2-Dichloroethene	20	40	80	200	600	2000	4000	8000		
trans-1,3-Dichloropropene	20	40	80	200	600	2000	4000	8000		
Trichloroethene	20	40	80	200	600	2000	4000	8000		
Trichlorofluoromethane	20	40	80	200	600	2000	4000	8000		
Vinyl chloride	20	40	80	200	600	2000	4000	8000		

¹Standards are spiked at all levels. A minimum of 5 points are used for each calibration model. Low points below the RL are routinely dropped and the high point might also be dropped for some analytes.

Table Bp-3: 5035FM_AK Calibration Levels (μg/Kg)¹ (Standards: MV-Supp Std and MV-2 Cleve)

Compound	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7	Level 8
1,1,1-Trifluoro-2,2- dichloroethane	20	40	80	200	600	2000	4000	8000
1,1,2-Trichloro-1,2,2- trifluoroethane	20	40	80	200	600	2000	4000	8000
1,2,3-Trimethylbenzene	20	40	80	200	600	2000	4000	8000
1,2-Dichloro-1,1,2,2- tetrafluoroethane	20	40	80	200	600	2000	4000	8000
1,2-Dichloro-1,1,2- trifluoroethane	20	40	80	200	600	2000	4000	8000
2-Chloroethy vinyl ether	20	40	80	200	600	2000	4000	8000
2-Nitropropane	20	40	80	200	600	2000	4000	8000
2-Pentanone	80	160	320	800	2400	8000	16000	32000
3-Chloro-1-propene (Allyl chloride)	20	40	80	200	600	2000	4000	8000
Carbon disulfide	20	40	80	200	600	2000	4000	8000
cis-1,4-dichloro-2-butene	20	40	80	200	600	2000	4000	8000
Cyclohexane	20	40	80	200	600	2000	4000	8000
Dichlorofluoromethane	20	40	80	200	600	2000	4000	8000
Ethyl acetate	40	80	160	400	1200	4000	8000	16000
Ethyl ether	20	40	80	200	600	2000	4000	8000

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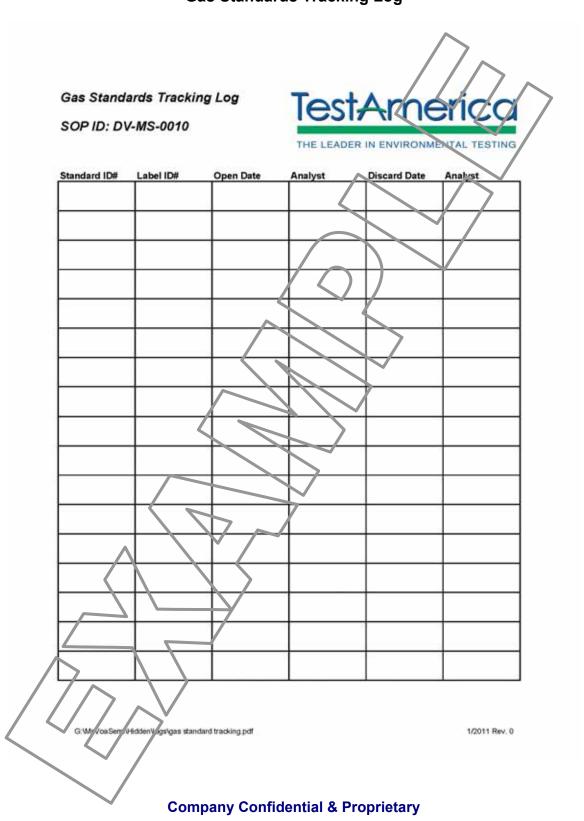
Table Bp-3: 5035FM_AK Calibration Levels (μg/Kg)¹ (Standards: MV-Supp Std and MV-2 Cleve)

Compound	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7	Level 8
Ethyl methacrylate	40	80	160	400	1200	4000	8000	16000
Ethylene oxide	2500	5000	10000	25000	75000	250000	500000	1000000
Hexane	20	40	80	200	600	2000	4000	8000
Isobutyl alcohol	400	800	1600	4000	12000	40000	80000	160000
Methyl acetate	100	200	400	1000	3000	10000	20000	40000
Methylcylcohexane	20	40	80	200	600	2000	4000	8000
Methyl methacrylate	40	80	160	400	1200	4000	8000	16000
Methyl tert-butyl ether (MTBE)	20	40	80	200	600	2000	4000	8000
Propene oxide	400	800	1600	4000	12000	40000	80000	160000
sec-Butyl alcohol	600	1200	2400	6000	18000	60000	120000	240000
tert-Amyl methyl ether	100	200	400	1000	3000	10000	20000	40000
tert-Butyl ethyl ether	100	200	400	1000	3000	10000	20000	40000
Tetrahydrofuran	40	80	160	400	1200	4000	8000	16000
trans-1,4-dichloro-2-butene	20	40	80	200	600	2000	4000	8000
Vinyl acetate	40	80	160	400	1200	4000	8000	16000

¹Standards are spiked at all levels. A minimum of 5 points are used for each calibration model. Low points below the RL are routinely dropped and the high point might also be dropped for some analytes.

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Attachment 1 Gas Standards Tracking Log





TestAmerica Denver

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Title: Concentration and Clean-up of Organic Extracts [SW-846, 3510C, 3520C,

3540C, 3546, 3550B, 3550C, 3620C, 3660B, 3665A, and EPA 600 Series Methods]

Approvals (Signature/Date):									
Susan Decker Technical Manager	/ <u>0//3/,,</u> Date	Adam Alban / 3 Oct / Adam Alban Date Health & Safety Manager / Coordinator							
John Morris Quality Assurance Manager	la 13 11 Date	Robert C. Hanisch Date Laboratory Director							

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1.0 Scope and Application

- 1.1 This standard operating procedure (SOP) provides instructions for the concentration, and if necessary, cleanup, of solvent extracts of organic compounds from water samples, soil samples, TCLP leachates, and SPLP leachates. This SOP is based on SW-846 Methods 3510C, 3520C, 3540C, 3546, 3550B, 3550C, 3620C, 3660B, 3665A, and EPA 600 Series methods.
- **1.2** The determinative methods and extraction methods used in conjunction with this procedure are listed in Attachment 1.

NOTE: This SOP does <u>not</u> include the concentration steps of extracts for Herbicides by method 8151A or 615. See DV-OP-0011 instead.

1.3 This procedure does not include the extraction steps. See the following SOPs for the applicable extraction procedures:

DV-OP-0006:	Extraction of Agu	leous Samples by	Separatory	Funnel, SW-

846 3510C and EPA 600 Series

DV-OP-0008: Extraction of Aqueous Samples by Continuous Liquid/Liquid

Extraction (CLLE) by Method SW-846 3520C and Methods

625 and 607

DV-OP-0015 Microwave Extraction of Solid Samples, SW-846 3546

DV-OP-0016: Ultrasonic Extraction of Solid Samples, SW-846 3550B and

3550C

DV-OP-0021: Extraction of Aqueous Samples by Continuous Liquid/Liquid

Extraction (CLLE) by Method SW-846 3520C for Low-Level

NDMA by GC/CI/MS/MS

DV-OP-0010: Soxhlet Extraction of Solid Samples, SW-846 3540C

DV-MS-0005. Liquid/Liquid Extraction (CLLE) by Method SW-846 3520C

Appendix II: for Extended List PAHS for CSLP.

2.0 Summary of Method

Sample extracts are concentrated to a specific final volume using an S-EVAP, N-EVAP, or Turbo-Vap. Some methods require a solvent exchange. If necessary, various clean-up techniques are performed before the extract is sent for analysis.

3.0 <u>Definitions</u>

3.1 Extraction Holding Time: The elapsed time expressed in days from the date of sample collection to the date the extraction starts. The holding time is tracked in the laboratory LIMS system, and is the primary basis of prioritizing work.

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3.2 Preparation Batch: A group of up to 20 samples that are of the same matrix and are processed together in the same extraction event using the same procedure and lots of reagents and standards.

- **3.3 Method Comments:** The Method Comments are used to communicate to the bench level chemists special requirements and instructions from the client. See WI-DV-0032
- 3.4 Quality Assurance Summary (QAS): Certain clients may require extensive specific project instructions or program QC, which are too lengthy to fit conveniently in the special instructions/Method Comments field in LIMS. In those situations, laboratory Project Managers describe the special requirements in a written QAS to address these requirements. QASs are posted on a public drive for easy accessibility by all lab employees. Normally QASs are introduced to analysts in an initial project kick-off meeting to be sure that the requirements are understood.

4.0 <u>Interferences</u>

Chemical and physical interferences may be encountered when analyzing samples using this method.

- 4.1 Method interferences may be caused by contaminants in solvents, reagents, glassware, and other processing apparatus that lead to discrete artifacts. All these materials must be routinely demonstrated to be free from interferences under conditions of the analysis by running laboratory method blanks as described in the Quality Control section. Specific selection of reagents may be required to avoid introduction of contaminants.
- **4.2** Visual interferences or anomalies (such as foaming, emulsions, odor, more than one layer of extract, etc.) must be documented.
- 4.3 The most common interference is laboratory contamination, which may arise from impure reagents, dirty glassware, improper sample transfers, dirty work areas, etc. Be aware of potential sources of contamination and take appropriate measures to minimize or avoid them.
- Due to the low reporting limits and the potential for contamination, the extracts that are to be analyzed for NDMA by GC/CI/MS/MS must be concentrated in glassware designated for that method. K-D flasks, concentrator tubes, and snyder columns will be clearly marked and segregated for this purpose.

5.0 Safety

Employees must abide by the policies and procedures in the Environmental Health and Safety Manual, Radiation Safety Manual and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, nitrile gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

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5.1 Specific Safety Concerns or Requirements

5.1.1 In order to limit the emission of methylene chloride, TestAmerica Denver uses a solvent recovery system. The system condenses and collects methylene chloride that has been evaporated off the sample extracts while on the S-EVAP.

- **5.1.1.1** Each analyst must inspect the system before using it to ensure the collection tubes are in good condition, the in-process tanks are not full, and the chiller is operating correctly.
- **5.1.1.2** While concentrating methylene chloride or methylene chloride / acetone extracts on the S-Evap, the analyst will use a timer set at 30 minute intervals to help remind the analyst to check the level of the solvent collected in the in-process tanks. This will be done to ensure that the tanks are not over-filled. A tank will not be filled more than 90%.
- **5.1.1.3** The solvent recovery system will never be used for the collection of ether due to the potential danger to analysts if the system were to fail during operation.

5.2 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table. A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material	Hazards	Exposure Limit (1)	Signs and Symptoms of Exposure
Acetonitrile	Flammable Irritant Poison	40ppm TWA	Exposure may cause cyanide poisoning resulting in reddening of the skin and eyes and pupil dilation. Effects of overexposure are often delayed due to the slow formation of cyanide ions in the body. May cause nose and throat irritation, flushing of the face, tightening of the chest. Also may cause headache, nausea, abdominal pain, convulsions, shock.
Hexane	Flammable Irritant	50ppm TWA	Causes irritation to eyes, skin and respiratory tract. Aspiration hazard if swallowed. Can enter lungs and cause damage. May cause nervous system effects. Breathing vapors may cause drowsiness and dizziness. Causes redness and pain to the skin and eyes.

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Material	Hazards	Exposure Limit (1)	Signs and Symptoms of Exposure
Methanol	Flammable Irritant Poison	200 ppm TWA	Methanol evaporates at room temperature. Inhalation, ingestion and/or eye and skin contact can all possibly cause light-headedness, nausea, headache, and drowsiness. Prolonged exposure can lead to permanent blindness.
Acetone	Flammable	1000 ppm-TWA	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache
Methylene Chloride	Irritant Carcinogen	25ppm TWA 125ppm STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting, and headache. Causes irritation, redness, and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.

⁽¹⁾ Always add acid to water to prevent violent reactions.

6.0 Equipment and Supplies

NOTE: All glassware used in this procedure is cleaned following SOP# DV-OP-0004.

In addition, the glassware is rinsed with methylene chloride immediately prior to

use.

NOTE: Due to the low reporting limits and the potential for contamination, the extracts that are to be analyzed for NDMA by GC/CI/MS/MS must be concentrated in

glassware designated for that method. K-D flasks, concentrator tubes, and snyder columns will be clearly marked and segregated for this purpose.

- Kuderna-Danish (K-D) flasks.
- Concentrator tubes for K-D flasks, un-graduated, approximately 10 mL.
- Concentrator tubes for K-D flasks, graduated at 1mL, calibration checked before use following the steps detailed in DV-QA-0008.
- Snyder columns, 3-ball with ground glass joints at top and bottom
- Manual, adjustable positive-displacement pipette and bottle-top re-pipettor, used to dispense 1 to 20 mL. Calibration is checked following the steps detailed in DV-QA-0008.
- Extract Storage Vials variety of sizes, clear and amber
- Pasteur pipettes 6 inch and 9 inch in length.
- Stem-less glass funnels
- Glass wool, baked at 400 □C for four hours.

⁽²⁾ Exposure limit refers to the OSHA regulatory exposure limit.

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 Boiling Chips – contaminant free, approximately 10/40 mesh Teflon®, PTFE. For concentrating extracts to a final volume greater than 1mL.

- Boiling Chips contaminant free, carborundum #12 granules, for concentrating extracts to a 1mL final volume. These boiling chips are sufficiently small as to not add any error to the 1mL final volume.
- Solvent Recovery System includes re-circulating chiller, set at 5□C, cooling condensers, Teflon® PTFE tubing and In-Process Tanks with quick-connect attachments
- S-Evap, thermostat controlled water bath
- N-Evap, thermostat controlled water bath with regulated nitrogen supply

7.0 Reagents and Standards

Reagents - All materials must be reagent grade or higher quality, unless otherwise specified

7.1 Methylene Chloride

Each lot of solvent is tested following CA-Q-S-001 or before it is put into use. QA personnel post the list of approved lots at solvent storage areas. For solvents packaged in CYCLETAINERS, that have not been previously tested per CA-Q-S-001, the first batch of samples prepared with a new lot of solvent is monitored and reported to the QA group per the instructions in CA-Q-S-001 DV-1. If any problems are identified, use of the solvent is suspended until further testing can be done and determines the solvent is acceptable.

7.2 Hexane

For solvents packaged in bottles, each lot of solvent is tested following CA-Q-S-001 before it is put into use. QA personnel post the list of approved lots at solvent storage areas. For solvents packaged in CYCLETAINERS, the first batch of samples prepared with a new lot of solvent is monitored and reported to the QA group per the instructions in CA-Q-S-001 DV-1. If any problems are identified, use of the solvent is suspended until further testing can be done and determines the solvent is acceptable.

7.3 Methanol, HPLC Grade

Each lot of solvent is tested following CA-Q-S-001 before it is put into use. QA personnel post the list of approved lots at solvent storage areas.

7.4 Acetone

Each lot of solvent is tested following CA-Q-S-001 before it is put into use. QA personnel post the list of approved lots at solvent storage areas.

7.5 Acetonitrile

Each lot of solvent is tested following CA-Q-S-001 DV-1 before it is put into use. QA personnel post the list of approved lots at solvent storage areas.

7.6 Baked Sodium Sulfate, 12-60 mesh

Heat sodium sulfate in a 400 □C oven for at least four hours. Each lot is tested as described in SOP CA-Q-S-001-DV-1

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7.7 Sulfuric Acid, Concentrated –

For use in PCB extract clean-up.

8.0 <u>Sample Collection, Preservation, Shipment and Storage</u>

Sample extracts waiting to be concentrated are stored refrigerated at $4^{\circ}\text{C} \pm 2^{\circ}\text{C}$ in glass bottles or flasks and capped with Teflon-lined lids or aluminum foil. Final sample extracts are stored in glass vials with Teflon-lined lids. See Table 3 for details on storage vial types. Final concentrated extracts for method 8270, 8270 SIM, and 8270 Best Practice are stored frozen at -10°C to -20°C, while all other extracts are stored refrigerated at $4^{\circ}\text{C} \pm 2^{\circ}\text{C}$.

Extracts have a holding time of 40 days from the date of extraction to the date of analysis.

9.0 Quality Control

9.1 The minimum quality controls (QC), acceptance criteria, and corrective actions are described in this section. When processing samples in the laboratory, use the LIMS QC program code and special instructions to determine specific QC requirements that apply.

The laboratory's standard QC requirements, the process of establishing control limits, and the use of control charts are described more completely in DV-QA-003P, Quality Assurance Program.

Specific QC requirements for Federal programs, e.g., Department of Defense (DoD) Department of Energy (DoE), AFCEE etc., are desribed in TestAmerica Denver policy DV-QA-024P, Requirements for Federal Programs.

Project-specific requirements can override the requirements presented in this section when there is a written agreement between the laboratory and the client, and the source of those requirements should be described in the project documents. Project-specific requirements are communicated to the analyst via special instructions in the LIMS.

Any QC result that fails to meet control criteria must be documented in a Nonconformance Memo (NCM). The NCM is approved by the supervisor and then automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group also receives NCMs by e-mail for tracking and trending purposes. The NCM process is described in more detail in SOP DV-QA-0031. This is in addition to the corrective actions described in the following sections.

9.2 Initial Performance Studies

Before analyzing samples, the laboratory must establish a method detection limit (MDL). In addition, an initial demonstration of capability (IDOC) must be performed by each analyst on the instrument he/she will be using. On-going proficiency must be demonstrated by each analyst on an annual basis. See Section 12 for more details on detection limit studies, initial demonstrations of capability, and analyst training and qualification.

9.3 Batch Definition

Batches are defined at the sample preparation stage. The batch is a set of up to 20 samples of the same matrix, plus required QC samples, processed using the same procedures and reagents within the same time period. Batches should be kept together through the whole analytical process as far as possible, but it is not mandatory to analyze prepared extracts on

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the same instrument or in the same sequence. The method blank must be run on each instrument that is used to analyze samples from the same preparation batch. See QC Policy DV-QA-003P for further details.

9.4 Method Blank (MB)

At least one method blank must be processed with each preparation batch. The method blank for batches of aqueous samples consists of reagent water, and for batches of soil samples, consists of Ottawa sand, both of which are free of any of the analyte(s) of interest. The method blank for batches of TCLP and SPLP leachates consists of leach fluid. The method blank is processed and analyzed just as if it were a field sample.

Acceptance Criteria: The result for the method blank must be less than the reporting limit for the analyte(s) of interest or less than 10% of the analyte concentration found in the associated samples, whichever is higher. Note that some programs (e.g., AFCEE, Navy, and USACE) require that the maximum blank concentration must be less than one-half of the reporting limit or less than 10% of the lowest sample concentration.

Corrective Action: If target analytes in the blank exceed the acceptance limits, an unacceptable method blank must be re-prepared and reanalyzed. If the analyte was <u>not</u> detected in the samples, then the data may be reported with qualifiers (check project requirements to be sure this is allowed) and it must be addressed in the project narrative.

9.5 Laboratory Control Sample (LCS)

At least one LCS must be processed with each preparation batch. For aqueous sample batches, the LCS consists of reagent water to which the analyte(s) of interest are added at known concentration. For soil sample batches, the LCS consists of Ottawa sand to which the analyte(s) of interest are added at a known concentration. For TCLP and SPLP leachates, the LCS consists of leach fluid to which the analyte(s) of interest are added at known concentration. The LCS is carried through the entire analytical procedure just as if it were a sample.

EPA Methods 608, 610, and 625 require a LCS at a 10% frequency. In other words, one LCS is required for a batch of 10 or less samples. A LCSD is required for a batch of 11 or more samples.

Acceptance Criteria: The recovery results for the LCS must fall within the established control limits. Control limits are set at \pm 3 standard deviations around the historical mean. Where required, project-specific limits may be used in place of historical limits. Current control limits are maintained in the LIMS.

When there are more than 11 analytes in the LCS, then NELAC allows a specified number of results to fall beyond the LCS control limit (3 standard deviations), but within the marginal exceedance (ME) limits, which are set at \pm 4 standard deviations around the mean of historical data. The number of marginal exceedances is based on the number of analytes in the LCS, as shown in the following table:

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# of Analytes in LCS	# of Allowed MEs
> 90	5
71 – 90	4
51 – 70	3
31 – 50	2
11 – 30	1
< 11	0

If more analytes exceed the LCS control limits than is allowed, or if any analyte exceeds the ME limits, the LCS fails and corrective action is necessary. Marginal exceedances must be random. If the same analyte repeatedly fails the LCS control limits, it is an indication of a systematic problem. The source of the error must be identified and corrective action taken.

Note: Marginal exceedances are not allowed for South Carolina work.

Corrective Action: If LCS recoveries are outside of the established control limits, the system is out of control and corrective action must occur. If recoveries are above the upper control limit and the analyte(s) of interest is not detected in samples, the data may be reported with qualifiers (check project requirements to be sure this is allowed) and it must be addressed in the project narrative. In other circumstances, the entire batch must be reprepared and reanalyzed.

9.6 Matrix Spike/Matrix Spike Duplicate (MS/MSD)

One MS/MSD pair must be processed with each preparation batch. A matrix spike (MS) is a field sample to which known concentrations of target analytes have been added. It is prepared in a manner similar to the LCS, but uses a real sample matrix in place of the blank matrix. A matrix spike duplicate (MSD) is a second aliquot of the same sample (spiked exactly as the MS) that is prepared and analyzed along with the sample and matrix spike. Some programs allow spikes to be reported for project-related samples only. Samples identified as field blanks cannot be used for the MS/MSD analysis.

EPA Methods 608, 610, and 625 require one matrix spike for every 10 samples. If the batch has more than 10 samples, then two matrix spikes must be performed. The two matrix spikes are to be performed on two different samples.

If insufficient sample volume is available for MS/MSD, an NCM must be written and a LCSD must be prepared.

Corrective Action: If analyte recovery or RPD falls outside the acceptance range, but the associated LCS recovery is in control, and all other QC criteria (e.g., continuing calibration verification) are met, then there is no evidence of analytical problems, and qualified results may be reported. The situation must be described in an NCM and in the final report case narrative. In other circumstances, the batch must be re-prepared and reanalyzed.

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9.7 Surrogate Spikes

Every calibration standard, field sample, and QC sample (i.e. method blank, LCS, LCSD, MS, and MSD) is spiked with surrogate compounds.

Acceptance Criteria: The recovery of each surrogate must fall within established statistical limits, which are set at \pm 3 standard deviations around the historical mean.

Corrective Action: If surrogate recoveries in the method blank are outside the established limits, verify calculations, standard solutions, and acceptable instrument performance. High surrogate recoveries in the blank might be acceptable if the surrogate recoveries for the field samples and other QC samples in the batch are acceptable. Low surrogate recoveries in the blank require re-preparation and reanalysis of the associated samples, unless sample surrogate recoveries are acceptable and targeted compounds are not detected.

If surrogate recoveries fail, verify calculations, standard solutions, and acceptable instrument performance. High recoveries may be due to a co-eluting matrix interference, which can be confirmed by examining the sample chromatogram. Low recoveries may be due to adsorption by the sample matrix (i.e., clay particles, peat or organic material in the sample). Recalculate the data and/or reanalyze the extract if the checks reveal a problem.

If matrix interference is not obvious from the initial analysis, it is necessary to re-prepare / reanalyze a sample only once to demonstrate that poor surrogate recovery is due to a matrix effect, as long as it can be shown that the analytical system was in control.

10.0 Procedure

10.1 One-time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using an NCM. The NCM is approved by the supervisor and then automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group also receives NCMs by e-mail for tracking and trending purposes. The NCM process is described in more detail in SOP DV-QA-0031. The NCM shall be filed in the project file and addressed in the case narrative.

10.2 Critical Procedural Considerations

- **10.2.1** As stated throughout this SOP, analysts must review Method Comments and any applicable QASs before starting work. This review is also documented on the Organic Extraction Checklist (see WI-DV-0009).
- 10.2.2 Analyst must focus on using clean technique throughout this procedure. Any parts or pipettes that come into direct contact with dirty surfaces should be cleaned or disposed of before coming into contact with the sample.
- **10.2.3** According to the type of sample and any cleanup procedures needed, different final solvents and volumes will be required. Refer to Attachment 3 for the appropriate final solvents and final volumes.

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10.3 Refer to Attachment 3 to determine if the extract is to be concentrated by the Kuderna-Danish / N-Evap method described in Section 10.4 and 10.5, or the Turbo-Vap method described in Section 10.6

- **10.4** Concentration by the Kuderna-Danish Method
 - 10.4.1 Refer to Attachment 3. If the extract is to be concentrated to a 1mL final volume, use a 1mL graduated concentrator tube. For extracts that are to be concentrated to any other final volume, use an un-graduated concentrator tube.
 - 10.4.2 Assemble the Kuderna-Danish concentrator by attaching the appropriate concentrator tube to the 500 mL K-D flask with a clip. Make sure the attachment is firm by twisting the concentrator tube at the joint while wearing cut-resistant gloves. Refer to Attachment 4 for configuration of the Kuderna-Danish concentrator.

NOTE: Due to the low reporting limits and the potential for contamination, the extracts that are to be analyzed for NDMA by GC/CI/MS/MS must be concentrated in glassware designated for that method. K-D flasks, concentrator tubes, and snyder columns will be clearly marked and segregated for this purpose.

- **10.4.3** Rinse the apparatus with methylene chloride. Discard the rinse solvent into the appropriate waste container. Care should be taken to ensure all surfaces of the glass are coated with solvent.
- **10.4.4** If the extract is to be concentrated to a 1mL final volume, add 2-3 carborundum granules to the K-D concentrator. If the extract is to be concentrated to a final volume greater than 1mL, add 1-2 Teflon® boiling chips to each K-D concentrator.
- 10.4.5 If the sample extracts have not been filtered through sodium sulfate at the time of extraction, or if the sample extract has visible water, then the extract must be dried at this point. Plug a glass funnel with baked glass wool and add baked sodium sulfate. Rinse the funnel and the sodium sulfate with methylene chloride and place it on top of the K-D. During the quantitative transfer in section 10.4.6 the extract will be filtered through the sodium sulfate.
- 10.4.6 Quantitatively transfer the sample extract to the K-D flask. Transfer the sample label to the K-D flask. Perform a quantitative transfer of the extract by rinsing the sample extract container with methylene chloride and adding the rinse solvent to the K-D. If the extract is being filtered through sodium sulfate, be sure to rinse the sodium sulfate well to ensure no target compounds are left on the sodium sulfate. Allow the solvent to drain from the sodium sulfate into the K-D flask then discard the sodium sulfate.
- **10.4.7** Turn a three-ball Snyder column upside down and rinse with methylene chloride, then rinse the bottom joint with methylene chloride. Attach the Snyder column to the top of the K-D concentrator as shown in Attachment

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4.

- **10.4.8** Place the K-D concentrator on a water bath so that the tip of the receiver tube is submerged. Refer to Attachment 3 for the correct water bath temperature. The water level should not reach the joint between the concentrator tube and the K-D flask.
- **10.4.9** For extracts that are methylene chloride or 50/50 methylene chloride/acetone, attach the solvent recovery system tube to the top of the Snyder column. At the appropriate rate of distillation, the balls will actively chatter but the chambers should not flood.

NOTE: For extracts for analysis for low-level NDMA by GC/CI/MS/MS, the solvent recovery system will not be used to avoid possible contamination.

NOTE: At this time, set a timer for no longer than 30 minutes as a reminder to check the in-process solvent tanks.

- 10.4.10 If the method does not require a solvent exchange, skip to Section 10.4.12. If the method requires a solvent exchange, continue on to Section 10.4.11.
- 10.4.11 If the method requires a solvent exchange at this time, detach the solvent recovery system tube from the top of the Snyder column and add the appropriate exchange solvent through the top of the Snyder column. The exchange solvent should be added when the extract has concentrated to a level that it forms a quarter-sized pool of solvent in the bottom of the K-D. Refer to Attachment 3 for details of exchange solvents and volumes. Mark the K-D flask and sample label to indicate the exchange has been performed. There is no need to re-attach the solvent recovery system at this time as the majority of the methylene chloride has already been evaporated and collected.
- 10.4.12 Continue to concentrate the sample on the water bath back down to 10-15 mL, or just below the K-D and concentrator tube joint. At this point the boiling sample is just barely splashing above the top of the receiver tube.
 - **NOTE:** It is very important not to concentrate to dryness as analytes will be lost. Also, some of the analyses, especially for 8270 and 8015, are especially temperature sensitive and the sample should be taken off the water bath as soon as possible to avoid losing analytes. Also the 8081 surrogate TCMX is also fairly volatile and can be lost if the extract is allowed to concentrate too low either before or after hexane exchange.
- **10.4.13** Remove the K-D concentrator from the water bath. Rinse the Snyder column down with a minimal amount of solvent. If the extract was exchanged, use the exchange solvent to perform the rinse, otherwise use methylene chloride.

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10.4.14 Allow the extract to cool to room temperature, about 10 minutes.

- 10.4.15 After the extract is allowed to cool, if the level of the extract is above the level of the concentrator tube joint, add a fresh boiling chip and return the K-D concentrator to the water bath.
- 10.4.16 After the extract is cool, remove the snyder column. Remove the clip holding the K-D flask and concentrator tube together. Use a Kim-wipe to dry the water off of the joint area so that water does not get into the extract. Remove the concentrator tube from the K-D flask and rinse the lower K-D flask joint into the concentrator tube with methylene chloride or the appropriate exchange solvent.
- **10.5** Nitrogen Evaporation (N-Evap) to Final Concentration.
 - **10.5.1** N-evap needles should be cleaned weekly by soaking overnight in methylene chloride. This is documented in the N-evap needle log-book.
 - **10.5.2** At the beginning of each shift, the N-evap needles should be wiped clean with a Kim-wipe soaked in methylene chloride to remove any potential contamination. If a needle comes in contact with an extract, then it needs to be cleaned before being used on the next extract.
 - 10.5.3 Place the concentrator tube on the nitrogen evaporator. The temperature of the water bath should be at least 5 °C below the boiling temperature of the solvent being evaporated (See Attachment 2). Lower the needle down to the sample so that a small dimple forms on the surface of the solvent. The stream of nitrogen should be gentle enough that it does not cause the extract to splash.
 - **10.5.4** During the course of the evaporation, rinse the sides of the concentrator tube with approximately 1 mL of clean solvent. The rinse should occur when the solvent gets close to the final volume. Concentrate the solvent to just below the final volume and remove from the nitrogen evaporator.
 - **10.5.5** Transfer the extract into the appropriate vial. Refer to Attachment 3 for the appropriate final volume and correct vial.
 - 10.5.5.1 If the extracts are to have a final volume of 1mL, they should be in 1mL graduated concentrator tubes. Using a Pasteur pipette, add the appropriate solvent to the tube until the extract meniscus reaches the 1mL gradation. Then using the Pasteur pipette transfer the extract to a labeled 2mL amber glass vial.
 - 10.5.5.2 For extracts with a final volume greater than 1mL, the vials should be calibrated using the manual, adjustable positive-displacement pipette or bottle-top re-pipettor. Pipette the correct volume of clean solvent into the vial and mark the bottom of the meniscus with a thin marker. Discard the solvent. Transfer the extract into the vial using a Pasteur pipette and rinse the concentrator tube with solvent. Transfer the rinse to the vial. Bring

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the meniscus of the solvent up to the marked line. Cap with a Teflon-lined cap.

NOTE: The final concentration and volume measurement steps are critical. Use care when concentrating and make certain that the final volume measurement is accurate.

NOTE: Some extracts might not concentrate down to the required final volume. If the extract is very dark and viscous, or an oil layer or precipitate starts to form, a higher final volume can be used. This should be documented in an NCM.

10.6 TurboVap Method

- **10.6.1** Turn on the TurboVap and adjust the water temperature to 40 °C. Turn the nitrogen supply on.
- **10.6.2** Switch the endpoint sensor to "Manual".
- **10.6.3** Adjust the water bath level. The water level should be above the extract level.
- 10.6.4 Turn on the nitrogen gas and adjust the gas pressure to approximately 12 psi. Lower pressure may be used if needed to prevent samples from splashing out of the TurboVap tubes.
- **10.6.5** Rinse the TurboVap tube with methylene chloride or the solvent the extract is in. Discard the waste.
- 10.6.6 Transfer the sample to the TurboVap tube. For 8141 soils extracted by soxhlet, dry the extract first by filtering through a funnel with baked sodium sulfate. Rinse the sample extract container with clean solvent and transfer to the TurboVap tube. Do not fill the TurboVap tubes over the fill line or approximately ¾ full.
- **10.6.7** Place the TurboVap tube into the TurboVap and turn on nitrogen to the position the tube is in.
- **10.6.8** Close the lid. You should be able to see the sample extracts swirling in the tubes.
 - **NOTE:** If the extract splashes when the nitrogen flow starts, transfer a portion of the extract back into the original extract container, or lower the gas pressure.
- 10.6.9 As the extract concentrates, transfer the remainder of the extract in to the appropriate Turbovap tube. Rinse the sample container with a few milliliters of methylene chloride or appropriate solvent and transfer to the Turbovap tube.
- **10.6.10** During the concentration rinse the Turbovap tube walls with a few

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milliliters of solvent 1 or 2 times.

10.6.11 If a solvent exchange is required, concentrate to about 5 mL and add the exchange solvent. After the exchange solvent is added, swirl the extract to make sure the extract is well mixed. Concentrate back down to slightly less than the appropriate volume. Refer to Attachment 3 for details of exchange solvents and final volumes.

- **10.6.12** Transfer the extract into the appropriate vial.
 - 10.6.12.1 Currently, the TurboVap is only used to concentrate extracts with final volumes greater than 1mL. Ask the QA Manager or the supervisor for guidance if a project requires a 1mL final volume by TurboVap.
 - 10.6.12.2 For extracts with a final volume greater than 1mL, the vials should be calibrated using the manual, adjustable pipette or bottle-top re-pipettor. Pipette the correct volume of clean solvent into the vial and mark the bottom of the meniscus with a thin marker. Discard the solvent. Transfer the extract to the vial using a Pasteur pipette and rinse the concentrator tube with solvent. Transfer the rinse to the vial. Bring the meniscus of the solvent up to the marked line. Cap with a Teflon-lined cap.
 - 10.6.12.3 Rinse the Turbovap tube with methylene chloride 2-3 times before washing. Turbovap tubes are not baked. They are cleaned in accordance with DV-OP-0004. If the Turbovap tubes need to be used again before they are dry, rinse with acetone to dry the Turbovap tube.

10.7 Cleanup Techniques

NOTE: If any sample in a batch requires a clean-up, the batch QC must also undergo the same clean-up technique.

10.7.1 Florisil Cartridge Cleanup

Florisil can be used to remove low-medium molecular weight polar hydrocarbon interfering compounds from pesticide extracts. The laboratory will use Florisil cleanups whenever water extracts have any color, whenever soil extracts have any color darker than a Post-It® Note, or whenever there is clear evidence of interferences, such as significant interfering peaks in the RT range for the target pesticide compounds or failing sample surrogate recoveries. Extracts that are to be analyzed for kepone will not be florisil cleaned, because florisil will remove kepone from the extract.

NOTE: Florisil cartridge performance checks are conducted for every lot of Florisil before use. Add 1.0 mL of the Florisil check solution described in Attachment 5 to a pre-rinsed Florisil cartridge. Following the procedure described below, load and elute the 1mL of check solution through the Florisil cartridge. Bring the final volume

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back down to 1.0 mL in hexane. The test sample must show 80-120 % recovery of all pesticide analytes with < 5% trichlorophenol recovery, and no peaks interfering with target compounds can be detected.

10.7.1.1 Clean the manifold and ports

Prior to each use, the top and underside of the manifold lid must be wiped down with hexane and a Kim-wipe to prevent any cross-contamination. The manifold ports must be dis-assembled and placed in a jar with fresh acetonitrile, in a sonication bath for a minimum of 30 minutes. The jar used in the soak and sonication of the ports must be replaced weekly to ensure it does not spread contamination. This is documented in the Organic Extraction Weekly Cleaning Logbook.

- **10.7.1.2** Place one Florisil cartridge into the vacuum manifold for each sample extract. Make sure all valves are closed.
- **10.7.1.3** Add 5 mL of hexane to each cartridge.
- **10.7.1.4** Slowly open the valves to allow a few drops of hexane to pass through, then close the valve and allow the hexane to soak the cartridge for at least 5 minutes.
- **10.7.1.5** Slowly open the valves again and allow the hexane to drain through the cartridge but close the valve when the solvent level is right above the glass frit. Do not allow the cartridges to go dry. If cartridges go dry, repeat the conditioning step.
- 10.7.1.6 Remove the manifold top and place one clean, labeled 16 \times 125 mm disposable glass test tube in each position for each of the samples. Replace the vacuum manifold top. Make sure that the solvent line from each cartridge is placed inside the appropriate tube.
- **10.7.1.7** Add exactly 2.0 mL of the concentrated extract to the appropriate Florisil cartridge. Turn the valve to the on position.
- **10.7.1.8** Allow the extract to gravity drip through the cartridge. The flow through the cartridges should be approximately 2 mL/minute.
- **10.7.1.9** Just before the Florisil cartridge goes, dry add 5 mL of hexane:acetone (90:10). Allow this to pass through the cartridge, then just before it goes dry again, add another 5 mL of hexane:acetone (90:10).
- **10.7.1.10** Allow the Florisil cartridge to go dry after the second addition of hexane:acetone (90:10). Turn the vacuum pump on after all of the cartridges have gone dry to recover any remaining solvent.
- **10.7.1.11** Remove the tubes from the vacuum manifold and

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concentrate them back down to 2.0 mL on the nitrogen evaporator. Transfer them to the appropriate vial. Discard the used cartridges.

10.7.2 Sulfur Removal

Sulfur can be removed by one of three methods: mercury, copper, or tetrabutylammonium sulfite (TBA), according to laboratory preference. If the sulfur concentration is such that crystallization occurs in the concentrated extract, centrifuge the extract to settle the crystals, and carefully draw off the sample extract with a disposable pipette, leaving the excess sulfur in the centrifuge tube. Transfer the extract to a clean concentrator tube before proceeding with further sulfur cleanup.

NOTE: Some programs (e.g., South Carolina) do not allow the use of elemental mercury. Copper or TBA will be used as an alternative.

10.7.2.1 Sulfur Removal with Elemental Mercury

NOTE: Use Mercury sparingly in order to minimize exposure and disposal costs.

- **10.7.2.1.1** Transfer approximately 2 mL of sample extract into a clean Teflon-sealed vial.
- **10.7.2.1.2** Add one to three drops of mercury to the extract vial and seal.
- **10.7.2.1.3** Shake well for 15-30 seconds. If prolonged shaking is required, use a mechanical shaker.
- **10.7.2.1.4** Remove the extract from the mercury using a disposable pipette and transfer to a clean vial.
- 10.7.2.1.5 If the mercury turns black, sulfur was present. Decant or pipette off the extract to a clean vial and repeat the procedure by adding one to three drops of fresh mercury. Do this until the mercury does not turn black.
- **10.7.2.1.6** If the extract is cloudy, filter the extract through a 1um disposable syringe filter.
- **10.7.2.1.7** Properly dispose of the mercury waste.
- **10.7.2.2** Sulfur Removal with Copper Powder

NOTE: This technique requires the copper powder to be very reactive, as demonstrated by a bright and shiny appearance. A pre-cleaned, activated copper may be purchased from a valid vendor. If manual preparation of reactive copper is performed, take care to remove all

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traces of acid in order to prevent degradation of some analytes.

- **10.7.2.2.1** Weigh out copper into a 20ml VOA VIAL assuming two grams of copper needed per sample.
- **10.7.2.2.2** Remove oxides by treating with 10% nitric acid.
- **10.7.2.2.3** Rinse the copper with DI organic-free water three times to remove all traces of acid.
- **10.7.2.2.4** Rinse the copper with acetone and dry under a stream of nitrogen.
- **10.7.2.2.5** Add approximately 2 grams of the copper powder to a 2ml vial with approximately 1ml of sample extract and shake vigorously on a mechanical shaker for at least one minute.
- **10.7.2.2.6** After phase separate, draw off extract and transfer to a clean vial.

10.7.3 Sulfuric Acid Cleanup

10.7.3.1 Add 1 mL of concentrated sulfuric acid to approximately 1 mL of sample extract in a Teflon capped vial.

CAUTION: There must be no water or acetone present in the extract or the reaction may shatter the sample container.

- **10.7.3.2** Vortex for about 5 seconds and allow to settle. (Centrifuge if necessary)
- **10.7.3.3** Remove the sample extract (top layer) from the acid using a Pasteur pipette and transfer to a clean vial.

CAUTION: It is not necessary to remove all the extract since the final volume is already determined. Transferring any amount of sulfuric acid along with the extract will result in extremely rapid degradation of the chromatographic column

- 10.7.3.4 If the sulfuric acid layer becomes highly colored after shaking with the sample extract, transfer the hexane extract to a clean vial and repeat the cleanup procedure until color is no longer being removed by the acid, or a maximum of 5 acid cleanups.
- **10.7.3.5** Properly dispose of the acid waste.

11.0 Calibration

Not applicable to this procedure. See the determinative methods for calibration of the analytical instrumentation.

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12.0 Method Performance

12.1 Method Detection Limit Study (MDL)

Before analyzing samples, the laboratory must establish a method detection limit (MDL). The laboratory also operates under programs that require instrument detection limits (IDLs). See DV-QA-005P, "Determination of Method Detection Limits", for more information on the method detection limit studies.

12.2 Demonstration of Capabilities

An initial demonstration of capability (IDOC) must be performed by each analyst. On-going proficiency must be demonstrated by each analyst on an annual basis. See SOP DV-QA-0024, "Employee Training", for more information on the IDOCs.

12.3 Training Requirements

The group/team leader has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use and who has the required experience. Further details concerning the training program are described in SOP DV-QA-0024.

13.0 Pollution Control

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

14.0 Waste Management

- 14.1 All waste will be disposed of in accordance with Federal, State, and local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this procedure, the policies in section 13, "Waste Management and Pollution Prevention", of the Environmental Health & Safety Manual, and DV-HS-001P, "Waste Management Plan."
- **14.2** The following waste streams are produced when this method is carried out:
 - **14.2.1** Methylene chloride Waste Stream B
 - 14.2.2 Flammable Solvents Waste Stream C
 - 14.2.3 Solid waste/sodium sulfate Waste Stream D
- **14.3** Radioactive waste, mixed waste, and potentially radioactive waste must be segregated from non-radioactive waste as appropriate. Contact the Waste Coordinator for proper management of these materials.

15.0 References / Cross-References

15.1 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, Third Edition and all promulgated updates, U.S. Environmental Protection Agency, Office of Solid Waste and Emergency Response, January 2005.

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- **15.1.1** Method 3510C, Separatory Funnel Liquid-Liquid Extraction, Revision 3, December 1996.
- **15.1.2** Method 3520C, Continuous Liquid-Liquid Extraction, Revision 3, December 1996.
- **15.1.3** Method 3550B, Ultrasonic Extraction, Revision 2, December 1996.
- **15.1.4** Method 3550C, Ultrasonic Extraction, Revision 3, February 2007.
- **15.1.5** Method 3540C, Soxhlet Extraction, Revision 3, December 1996.
- **15.1.6** Method 3546, Microwave Extraction, Revision 0, February 2006.
- **15.1.7** Method 3620C, Florisil Cleanup, Revision 3, February 2007.
- 15.1.8 Method 3660B, Sulfur Cleanup, Revision 2, December 1996.
- **15.1.9** Method 3665A, Sulfuric Acid/Permagante Cleanup, Revision 1, December 1996.
- **15.2** Code of Federal Regulations, Title 40 Protection of the Environment, Part 136 Guidelines Establishing Test Procedures for the Analysis of Pollutants, Appendix A Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater
 - **15.2.1** Method 608, Organochlorine Pesticides and PCBs.
 - **15.2.2** Method 610, Polynuclear Aromatic Hydrocarbons.
 - **15.2.3** Method 614, The Determination of Organophosphorus Pesticides in Municipal and Industrial Wastewater
 - **15.2.4** Method 625, Base/Neutrals and Acids.

16.0 <u>Method Modifications:</u>

16.1 Method 3665A calls for the clean-up to be performed using 1:1 Sulfuric Acid:H2O. This procedure calls for the clean-up to be performed using concentrated sulfuric acid.

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17.0 Attachments

Attachment 1: Determinative and Extraction Methods Used in Conjunction with this SOP.

Attachment 2: Boiling Points of Solvents Attachment 3: Concentration Summary

Attachment 4: Kuderna-Danish Concentrator

Attachment 5: Florisil Check Solution

18.0 Revision History

Revision 6.0 dated 14 October 2011

- The procedure was revised to remove instructions on how to concentrate and clean up extract for method 8070 and 607. TestAmerica Denver no longer supports these methods.
- Section 1.3 was corrected to give the correct SOP number to Extraction of Aqueous Samples by Continuous Liquid/Liquid Extraction (CLLE) by Method SW-846 3520C for Low-Level NDMA by GC/CI/MS/MS.
- Section 7.5 was revised to state acetonitrile is tested before use. Previously this solvent was not tested before use.
- The procedure was revised to include instructions that all extracts for analysis by method 8081, 8082, or 608 to be hexane exchanged only after concentration on the S-Evap. Previously the SOP instructed analysts to add the hexane exchange before the S-Evap for extracts that were concentrated by microwave extraction. This resulted in poor hexane exchanges, therefore the extracts are now concentrated before the exchange.
- The procedure was revised to instruct analysts not to use the solvent recovery system when concentrating samples for analysis of low-level NDMA by GC/CI/MS/MS. This was done to eliminate a possible source of contamination in this ppt level analysis.
- The procedure was revised to instruct analysts to use concentrated sulfuric acid in the acid clean up of PCB extracts.
- The procedure was revised to clarify the exact steps used in the sulfur removal with mercury.
- Revision 5 dated 07/20/10
 - Note added to section 9.5 to not allow marginal exceedances for South Carolina work
 - Updated to reflect changes to the LIMS system.
 - Updated Attachment 1 and Section 1.3 to include the most recent extraction and analysis SOPs.
 - Added procedures to concentrate microwave extracts by K-D.
- Revision 4, dated 26 August 2009
 - Added instructions on the concentration of extracts from microwave extraction, SW846 3546.
 - Added clarification that the solvent recovery system is only to be used with extracts containing methylene chloride.
 - Added instructions on the use of 1mL graduated concentrator tubes to determine 1mL final volumes.
 - Changed the required temperature of the re-circulating chiller used in the solvent recovery system from 10°C to 8°C.

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 Added instructions on how to properly clean the manifold and valves used in florisil clean-up.

- o Added guidance on when samples should be taken through the florisil clean-up.
- o Change to the use of 1:1 Sulfuric Acid in the clean-up procedure.
- Revision 3.1, dated 10 October 2008
 - o Added references to method 3550C throughout SOP.
- Revision 3, dated 25 April 2008
 - o Integration for Testamerica and STL operations

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Attachment 1.

Determinative and Extraction Methods Used in Conjunction with this SOP

Method	Determinative	Determinative	Extraction Method	Extraction
Description	Method	Method SOP		Method SOP
Diesel Range Organics & Jet Fuels	SW-846 8015B, 8015C, California LUFT Method, & AK102 & AK103, NW- TPH, OK DRO	DV-GC-0002 DV-GC-0027	WATER: SW-846 3510C, AK102, AK103,NW-TPH, OK DRO SOIL: SW-846 3550B/C AK102, AK103 AK102, AK103,NW- TPH, OK DRO	WATER: DV-OP-0006 SOIL: DV-OP-0016
Chlorinated Pesticides	SW-846 8081A, 8081B & EPA Method 608	DV-GC-0020 DV-GC-0016 DV-GC-0026	WATER: SW-846 3510C SOIL: SW-846 3550B/C or SW- 846 3546	WATER: DV-OP-0006 SOIL: DV-OP-0016 or DV-OP-0015
Polychlorinated Biphenyls	SW-846 8082, 8082A EPA Method 608	DV-GC-0021 DV-GC-0016 DV-GC-0030	WATER: SW-846 3510C SOIL: SW-846 3550B/C or SW- 846 3546	WATER: DV-OP-0006 SOIL: DV-OP-0016 or DV-OP-0015
Organo- phosphorus Pesticides	SW-846 8141A, 8141B, & EPA Method 614	DV-GC-0017	WATER: SW-846 3510C SOIL: SW-846 3540C	WATER: DV-OP-0006 SOIL: DV-OP-0010
Polynuclear Aromatic Hydrocarbons	SW-846 8310 & EPA Method 610	DV-LC-0009	WATER: SW-846 3510C SOIL: SW-846 3550B/C	WATER: DV-OP-0006 SOIL: DV-OP-0016
Semi-volatiles by GC/MS	SW-846 8270C, 8270D & EPA 625	DV-MS-0011 DV-MS-0012	WATER: SW-846 3510C or 3520C SOIL: SW-846 3550B/C	WATER: DV-OP-0006 or DV-OP-0008 SOIL: DV-OP-0016
Low-Level Semi- Volatiles by GC/MS	SW-846 8270C	DV-MS-0011	WATER: SW-846 3520C	WATER: DV-OP-0008
Polynuclear Aromatic Hydrocarbons by GC/MS SIM	SW-846 8270C SIM	DV-MS-0002	WATER: SW-846 3520C SOIL: SW-846 3550B/C or SW- 846 3546	WATER: DV-OP-0008 SOIL: DV-OP-0016 or DV-OP-0015
n- Nitrosodimethyla mine by GC/CI/MS/MS	SOP	DV-LC-0019	WATER: SW-846 3520C SOIL: SW-846 3550B/C	WATER: DV-OP-0021 SOIL: DV-OP-0016
Extended List PAHs by GC/MS SIM for CSLP and Full Scan	SW-846 8270C	DV-MS-0005	WATER: SW-846 3520C	WATER: DV-MS-0005, Appendix II

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Attachment 2.

Boiling Points of Solvents

Solvent	Boiling Point (°C)
Methylene chloride	40
Acetone	56
Hexane	69
Methanol	65
Acetonitrile	82

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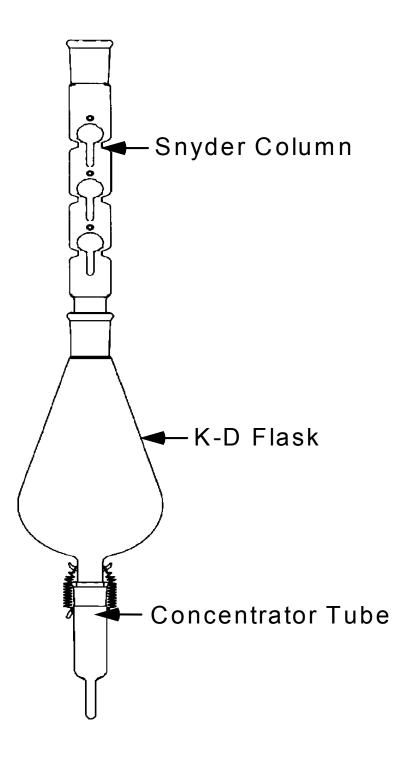
Attachment 3 - Concentration Summary (This table is included in controlled Work Instruction WI-DV-009)

Concentration Summary								
Analytical Method	Matrix	Extraction Solvent	Bath Temp (C)	Exchange Solvent	Exchange Vol. (mL)	Final Vol. (uL)	Vial Type	Cleanup Steps
625 8270C 8270D 8270_AFCEE 8270_DoD 8270C_UTS 8270C_FS_CSLP 8270C_SIM 8270D_SIM 8270_SIM_AFCEE 8270_SIM_DoD 8270C_SIM_CSLP 8270C_SIM_LL	Soil	MeCl ₂ :Acetone	S-Evap 88			1000	2 mL amber	
	Water and Leachate	MeCl ₂	S-Evap 84			1000	2 mL amber	
8270C_LL	Water	MeCl ₂	S-Evap 84			2000	4 mL amber	
8015B_DRO 8015C_DRO 8015D_DRO AK102_103 NWTPH_Dx Okla_DRO 8015B_Terp	Water or Soil	MeCl ₂	S-Evap 84			1000	2 mL amber	
NDMA_CIMSMS	Soil/Water	MeCl ₂	S-Evap 84			1000	2 mL amber	Designated glass!
608 8081A 8081B 8082 8082A	Soil by 3550C or 3546	MeCl ₂ :Acetone	S-Evap 88	Hexane	50	10,000	12 mL clear	Florisil if needed
	Soil by 3550C_LL or 3546_LL	MeCl ₂ :Acetone	S-Evap 88	Hexane	50	5000	12 mL clear	Florisil if needed
	Wipes by 3550C or 3546	Hexane	S-Evap 88			10,000	12mL clear	Florisil if needed
	Water by 3510C	MeCl2	S-Evap 88	Hexane	50	10,000	12 mL clear	Florisil if needed.
	Water by 3510C_LL	MeCl ₂	S-Evap 88	Hexane	50	1000	12 mL clear	
610 8310	Soil	MeCl ₂ :Acetone	S-Evap 88	ACN	10	4000	4 mL amber	
	Water	MeCl ₂	S-Evap 88	ACN	10	1000	2 mL amber	
614 8141A 8141B	Soil	MeCl ₂ :Acetone	Turbo-Vap 40	Hexane	50	2000	4 mL amber	
	Water	MeCl ₂	Turbo-Vap 40	Hexane	50	2000	4 mL amber	
8321A_Herb	Soil	Ether/Acetone	Turbo-Vap 40	ACN	2	5000	8 mL amber	Enter in 10mL Vf

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Attachment 4.

Kuderna-Danish Concentrator



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Attachment 5.

Florisil Check Solution Prepared in Hexane

Compound	Concentration
2,4,5-Trichlorophenol	0.1ug/mL
Alpha-BHC	0.05ug/mL
Alpha-Chlordane	0.05ug/mL
Aldrin	0.05ug/mL
Beta-BHC	0.05ug/mL
Dieldrin	0.05ug/mL
Endosulfan I	0.05ug/mL
Endosulfan II	0.05ug/mL
Endosulfan sulfate	0.05ug/mL
Endrin	0.05ug/mL
Endrin Aldehyde	0.05ug/mL
Endrin Ketone	0.05ug/mL
Gamma-BHC	0.05ug/mL
Gamma-Chlordane	0.05ug/mL
Heptachlor	0.05ug/mL
Heptachlor expoxide	0.05ug/mL
Methoxychlor	0.05ug/mL
4,4-DDD	0.05ug/mL
4,4-DDE	0.05ug/mL
4,4-DDT	0.05ug/mL



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SOP No. DV-OP-0010, Rev. 5 Effective Date: 7/13/12

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Title: Soxhlet Extraction of Solid Samples [SW-846 3540C]

Approvals (Signature/Date): Susan Oster Date Adam Alban Date				
তির্বাহি Adam Alban পুটারিছ বিজ্ঞান প্রত্তিক বিজ্ঞান প্রত্তিক বিজ্ঞান প্রত্তিক বিজ্ঞান বিজ্ঞা				
John Morris Date Robert C. Hanisch Date Quality Assurance Manager Laboratory Director				
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1.0 Scope and Application

- 1.1 This SOP is applicable to the solvent extraction of organic compounds from solid samples using a Soxhlet extractor. This is based on SW-846 Method 3540C.
- **1.2** The determinative methods used in conjunction with this procedure are listed in Attachment 1. This extraction procedure may be used for additional methods when appropriate spiking mixtures are used.
- 1.3 This procedure does not include the concentration and cleanup steps. See SOP # DV-OP-0007, "Organic Extract Concentration and Cleanup", for those details.
- **1.4** Determination of percent moisture described in the source method is performed according to SOP # DV-WC-0023 and is not included in this procedure.

2.0 Summary of Method

A measured weight of sample, typically 30 g, is mixed with anhydrous sodium sulfate to form a free flowing mixture. This mixture is placed into a Soxhlet extractor and extracted for 18 to 24 hours.

3.0 <u>Definitions</u>

- **3.1** Extraction Holding Time The elapsed time expressed in days from the date of sample collection to the date the extraction starts. The holding time is tracked in the laboratory LIMS system, and is the primary basis of prioritizing work.
- **3.2** Preparation Batch A group of up to 20 samples that are of the same matrix and are processed together in the same extraction event using the same procedure and lots of reagents and standards.
- 3.3 Method Comments The Method Comments are used to communicate to the bench level chemists special requirements and instructions from the client. See WI-DV-0032.
- Quality Assurance Summary (QAS) Certain clients may require extensive specific project instructions or program QC, which are too lengthy to fit conveniently in the special instructions/client requirements field in the LIMS. In those situations, laboratory Project Managers describe the special requirements in a written QAS. Each QAS is posted on a public drive for easy accessibility by all lab employees. Normally a QAS is introduced to analysts in an initial project kick-off meeting to be sure that the requirements are understood.
- 3.5 Aliquot A part which is a definite fraction of a whole; as aliquot samples for testing or analysis. "Aliquot" is also used as a verb meaning to take all or part of a sample for preparation, extraction, and/or analysis.

4.0 Interferences

4.1 Chemical and physical interferences may be encountered when analyzing samples using this method.

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4.2 It is very important that the Soxhlet extractor refluxes correctly. The extractor body should fill, drain, and fill again, completing the cycle 4-6 times an hour. If the extraction does not follow this cycle, but instead the solvent continuously flows through the soil sample and back into the boiling flask the extraction might not be as efficient.

- 4.3 Method interferences may be caused by contaminants in solvents, reagents, glassware, and other processing apparatus that lead to discrete artifacts. All these materials must be routinely demonstrated to be free from interferences under conditions of the analysis by running laboratory method blanks as described in the Quality Control section. Specific selection of reagents may be required to avoid introduction of contaminants.
- **4.4** Visual interferences or anomalies (such as foaming, emulsions, odor, etc.) must be documented.
- **4.5** The most common interference is laboratory contamination, which may arise from impure reagents, dirty glassware, improper sample transfers, dirty work areas, etc. Be aware of potential sources of contamination and take appropriate measures to minimize or avoid them.
- **4.6** Soap residue may cause degradation of certain analytes such as organophosphorus pesticides. Careful rinsing of glassware according to SOP DV-OP-0004 is required.

5.0 Safety

Employees must abide by the policies and procedures in the Environmental Health and Safety Manual, Radiation Safety Manual and this document.

This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, nitrile gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1 Specific Safety Concerns or Requirements

- **5.1.1** Eye protection that satisfies ANSI Z87.1, laboratory coat, and nitrile gloves must be worn while handling samples, standards, solvents, and reagents. Disposable gloves that have been contaminated must be removed and discarded; non-disposable gloves must be cleaned immediately.
- 5.1.2 Exposure to chemicals must be maintained as low as reasonably achievable; therefore, unless they are known to be non-hazardous, all samples must be opened, transferred, and prepared in a fume hood, or under other means of mechanical ventilation. Solvent and waste containers will be kept closed unless transfers are being made.
- **5.1.3** The preparation of standards and reagents will be conducted in a fume hood with the sash closed as far as the operation will permit.

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5.1.4 All work must be stopped in the event of a known or potential compromise to the health and safety of a TestAmerica associate. The situation must be reported immediately to a laboratory supervisor.

5.2 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating.

NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the Material Safety Data Sheet (MSDS) for each of the materials listed in the table.

A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Materials with Serious or Significant Hazard Rating

Material	Hazards	Exposure Limit (1)	Signs and Symptoms of Exposure		
Methylene Chloride	Carcinogen Irritant	25 ppm TWA 125 ppm STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting, and headache. Causes irritation, redness, and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.		
Acetone	Flammable	1000 ppm-TWA	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache.		
(1) Exposure limit refers to the OSHA regulatory exposure limit.					

6.0 **Equipment and Supplies**

6.1 Extraction Equipment

- Soxhlet Extractor with ground glass joints
- 250-mL boiling flask with ground glass joint
- Cooling Condensers.
- Vacuum pump.
- Heating Mantle, Rheostat controlled, or Hotplate with temperature control. If hotplates are used then a metal cup must also be used to even heat the boiling flasks.
- Balance >1400-g capacity, accurate to ±0.1 g, calibrated daily per SOP DV-QA-0014
- Pipetter with disposable 1.0-mL tips calibrated daily per SOP DV-QA-0008.

6.2 Supplies

- Boiling Chips Contaminant free, approximately 10/40 mesh, Teflon®, PTFE, carbide, or equivalent.
- Glass wool Baked at 400°C for at least four hours.
- Cellulose Extraction Thimble
- Beakers thick walled, 400 mL

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- Aluminum Foil.
- Wood tongue depressors, metal spatulas.
- Filter flask and vacuum pump to filter extracts with visible sediment.

7.0 Reagents and Standards

- **7.1 Reagents:** All materials must be reagent grade or higher quality, unless otherwise specified
 - 7.1.1 Methylene chloride Each lot of solvent is tested following SOP# CA-Q-S-001 or CA-Q-S-001 DV-1 before it is put into use. QA personnel post the list of approved lots at solvent storage areas.
 - 7.1.2 Acetone Each lot of solvent is tested following SOP# CA-Q-S-001 or CA-Q-S-001 DV-1 before it is put into use. QA personnel post the list of approved lots at solvent storage areas.
 - 7.1.3 1:1 Methylene Chloride: Acetone Blend Purchased ready to use in CYCLETAINERS. Each lot of solvent is tested following SOP# CA-Q-S-001 or CA-Q-S-001 DV-1 before it is put into use. QA personnel post the list of approved lots at solvent storage areas. If pre-mixed solvent is not available, solvent can be prepared by combining equal portions of methylene chloride and acetone as described in Sections 7.1.1 and 7.1.2
 - **7.1.4** Baked Sodium Sulfate, 12-60 mesh Heat sodium sulfate in a 400°C oven for at least four hours. Each lot is tested following CA-Q-S-001 DV-1 before it is put into use. QA personnel post the list of approved lots at solvent storage areas.
 - 7.1.5 Baked Ottawa Sand Heat Ottawa sand in a 400°C oven for at least four hours.

7.2 Standards

7.2.1 Please reference DV-OP-0020 for information regarding the surrogate and spike standards used in this procedure.

8.0 Sample Collection, Preservation, Shipment and Storage

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time ¹	SW-846 Reference
Soils	Glass	30 grams	Cool, 0-6 °C	14 Days	8141A
Soils	Glass	30 grams	Cool, 0-6 °C	7 Days	8141B

¹Exclusive of analysis.

[•] If the holding time is exceeded, a Nonconformance Memo (NCM) must be prepared and the client notified immediately.

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9.0 **Quality Control**

- 9.1 The minimum quality controls (QC), acceptance criteria, and corrective actions are described in this section. When processing samples in the laboratory, use the Method Comments to determine specific QC requirements that apply. See WI-DV-0032 for more information on Method Comments.
- **9.2** The laboratory's standard QC requirements, the process of establishing control limits, and the use of control charts are described more completely in DV-QA-003P, Quality Assurance Program.
- 9.3 Specific QC requirements for Federal programs, e.g., Department of Defense (DoD) Department of Energy (DoE), AFCEE etc., are described in TestAmerica Denver policy DV-QA-024P, Requirements for Federal Programs.
- 9.4 Project-specific requirements can override the requirements presented in this section when there is a written agreement between the laboratory and the client, and the source of those requirements should be described in the project documents. Project-specific requirements are communicated to the analyst via special instructions in the LIMS.
- 9.5 Any QC result that fails to meet control criteria must be documented in a Nonconformance Memo (NCM). The NCM is approved by the supervisor and then automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The NCM process is described in more detail in SOP DV-QA-0031. This is in addition to the corrective actions described in the following sections.

9.6 Initial Performance Studies

Before analyzing samples, the laboratory must establish a method detection limit (MDL). In addition, an initial demonstration of capability (IDOC) must be performed by each analyst on the instrument he/she will be using. On-going proficiency must be demonstrated by each analyst on an annual basis. See Section 13 for more details on detection limit studies, initial demonstrations of capability, and analyst training and qualification.

9.7 Batch Definition

Batches are defined at the sample preparation stage. The batch is a set of up to 20 samples of the same matrix, plus required QC samples, processed using the same procedures and reagents within the same time period. Batches should be kept together through the whole analytical process as far as possible, but it is not mandatory to analyze prepared extracts on the same instrument or in the same sequence. The method blank must be run on each instrument that is used to analyze samples from the same preparation batch. See QC Policy DV-QA-003P for further details.

9.8 Method Blank (MB)

At least one method blank must be processed with each preparation batch. The method blank is processed and analyzed just as if it were a field sample.

The method blank for batches of soil samples consists of 30 g of Ottawa sand free of any of the analyte(s) of interest.

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Acceptance Criteria: The result for the method blank must be less than one-half the reporting limit for the analyte(s) of interest or less than 10% of the analyte concentration found in the associated samples, whichever is higher. Note that some programs (e.g., AFCEE, Navy, and USACE) require that the maximum blank concentration must be less than one-half of the reporting limit or less than 10% of the lowest sample concentration.

Corrective Action: If target analytes in the blank exceed the acceptance limits, an unacceptable method blank must be re-prepared and reanalyzed. If the analyte was <u>not</u> detected in the samples, then the data may be reported with qualifiers (check project requirements to be sure this is allowed) and it must be addressed in the project narrative.

9.9 Laboratory Control Sample (LCS)

At least one LCS must be processed with each preparation batch. The LCS is carried through the entire analytical procedure just as if it were a sample.

For soil sample batches, the LCS consists of 30 g of Ottawa sand to which the analyte(s) of interest are added at known concentration.

<u>Acceptance Criteria</u>: The recovery results for the LCS must fall within the established control limits. Control limits are set at \pm 3 standard deviations around the historical mean. Where required, project-specific limits may be used in place of historical limits. Current control limits are maintained in the LIMS.

When there are more than 11 analytes in the LCS, then NELAC allows a specified number of results to fall beyond the LCS control limit (3 standard deviations), but within the marginal exceedance (ME) limits, which are set at \pm 4 standard deviations around the mean of historical data. The number of marginal exceedances is based on the number of analytes in the LCS, as shown in the following table:

# of Analytes in LCS	# of Allowed MEs
> 90	5
71 – 90	4
51 – 70	3
31 – 50	2
11 – 30	1
< 11	0

If more analytes exceed the LCS control limits than is allowed, or if any analyte exceeds the ME limits, the LCS fails and corrective action is necessary. Marginal exceedances must be random. If the same analyte repeatedly fails the LCS control limits, it is an indication of a systematic problem. The source of the error must be identified and corrective action taken.

<u>Corrective Action</u>: If LCS recoveries are outside of the established control limits, the system is out of control and corrective action must occur. If recoveries are above the upper control limit and the analyte(s) of interest is(are) not detected in samples, the data may be reported with qualifiers (check project requirements to be sure this is allowed) and it must be addressed in the project narrative. In other circumstances, the entire batch must be reprepared and reanalyzed.

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9.10 Matrix Spike/Matrix Spike Duplicate (MS/MSD)

One MS/MSD pair must be processed with each preparation batch. A matrix spike (MS) is a field sample to which known concentrations of target analytes have been added. It is prepared in a manner similar to the LCS, but uses a real sample matrix in place of the blank matrix. A matrix spike duplicate (MSD) is a second aliquot of the same sample (spiked exactly as the MS) that is prepared and analyzed along with the sample and matrix spike. Some programs allow spikes to be reported for project-related samples only. Samples identified as field blanks cannot be used for the MS/MSD analysis.

If insufficient sample volume is available for MS/MSD, an NCM must be written and a LCSD must be prepared.

<u>Corrective Action</u>: If analyte recovery or RPD falls outside the acceptance range, but the associated LCS recovery is in control, and all other QC criteria (e.g., continuing calibration verification) are met, then there is no evidence of analytical problems, and qualified results may be reported. The situation must be described in an NCM and in the final report case narrative. In other circumstances, the batch must be re-prepared and reanalyzed.

9.11 Surrogate Spikes

Every calibration standard, field sample, and QC sample (i.e. method blank, LCS, LCSD, MS, and MSD) is spiked with surrogate compounds.

<u>Acceptance Criteria:</u> The recovery of each surrogate must fall within established statistical limits, which are set at \pm 3 standard deviations around the historical mean.

<u>Corrective Action</u>: If surrogate recoveries in the method blank are outside the established limits, verify calculations, standard solutions, and acceptable instrument performance. High surrogate recoveries in the blank might be acceptable if the surrogate recoveries for the field samples and other QC samples in the batch are acceptable. Low surrogate recoveries in the blank require re-preparation and reanalysis of the associated samples, unless sample surrogate recoveries are acceptable and targeted compounds are not detected.

If surrogate recoveries fail, verify calculations, standard solutions, and acceptable instrument performance. High recoveries may be due to a co-eluting matrix interference, which can be confirmed by examining the sample chromatogram. Low recoveries may be due to adsorption by the sample matrix (i.e., clay particles, peat or organic material in the sample). Recalculate the data and/or reanalyze the extract if the checks reveal a problem.

If matrix interference is not obvious from the initial analysis, it is necessary to re-prepare / reanalyze a sample only once to demonstrate that poor surrogate recovery is due to a matrix effect, as long as it can be shown that the analytical system was in control.

10.0 Procedure

10.1 One time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance Memo (NCM) that is approved by a Technical Specialist and the QA Manager. If contractually required, the client shall be

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notified. The NCM shall be filed in the project file. See SOP# DV-QA-0031 for further details concerning the NCM process.

10.2 Critical Procedural Considerations

- 10.2.1 As stated throughout this SOP, analysts must review Method Comments and any applicable QASs before starting work. This review is also documented on the Organic Extraction Checklists (see Work Instruction WI-DV-009).
- 10.2.2 The analyst must focus on using clean technique throughout this procedure. Any parts or pipettes that come into direct contact with dirty surfaces or any beaker or media bottle other than the designated one should be cleaned or disposed of before coming into contact with the sample.

10.3 Clean the Glassware Immediately Before Use.

- **10.3.1** Rinse 400-mL thick-walled beakers with methylene chloride.
- **10.3.2** Rinse 250-mL boiling flasks with methylene chloride or a 1:1 mixture of methylene chloride and acetone. Discard the rinse and place 1 to 5 boiling chips in the bottom of the flask. Fill the flask just below the neck with approximately 250 mL of a 1:1 mixture of methylene chloride and acetone.
- **10.3.3** Rinse the Soxhlet extractor with methylene chloride taking care that the solvent comes in contact with all inside surfaces of the glassware.

10.4 Aliquot Samples

- **10.4.1** Follow the procedure for mixing and homogenizing samples in SOP# DV-QA-0023, "Subsampling."
- **10.4.2** Label a 400-mL beaker with the sample ID, method, and batch number.
- 10.4.3 Weigh 30 g 33 g of sample into the labeled beaker. For each method blank and LCS sample, weigh 30 g 33 g of baked Ottawa sand into labeled beakers.
- **10.4.4** Record the weight to the nearest 0.1 g directly into the LIMS or hand record on the benchsheet printed from LIMS. (See Attachment 2 for an example benchsheet).
- Mix the weighed sample with a wooden tongue depressor or metal spatula. Add enough sodium sulfate so the sample becomes free flowing. If the sample is not free flowing, the extraction efficiency may be reduced. Although the baked Ottawa sand used in the method blank and the LCS is already free-flowing, sodium sulfate will be added to these QC samples as well. This is done to demonstrate that the sodium sulfate does not interfere with the procedure or contaminate the samples.
- 10.4.6 Rinse a cellulose extraction thimble with methylene chloride and transfer the sample and sodium sulfate mixture into a cellulose extraction thimble. Fill the thimble with sodium sulfate up to ¼ inch below the top of the thimble. Carefully place the extraction thimble inside the rinsed soxhlet extractor. If thimbles are

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not available, glass wool can be used instead. Follow steps 10.4.6.1 and 10.4.6.2 in the absence of a thimble.

- 10.4.6.1 Take a thin piece of glass wool and place it inside the Soxhlet extractor body so that it covers the drain line. You can use a small amount of methylene chloride to wet the glass wool so that it stays in place. It is important that the glass wool prevent solids from entering the drain line, but it must not hinder the solvent from draining out of the extractor. That is why it is important to use a thin piece of glass wool.
- **10.4.6.2** Being careful not to disturb the glass wool, add a thin layer of sodium sulfate (approximately ¾" to 1" thick) to the bottom of the soxhlet, then transfer the sample to the extractor body. Once the entire sample has been transferred, cover the sample with a layer of sodium sulfate. The solids in the extractor body should not be higher than the peak of the drain line. Also if the solids are too low, the solvent level in the round bottom will go too low before the cycling begins.

10.5 Assemble the Soxhlet Apparatus

10.5.1 Attach the solvent-filled boiling flask to the bottom of the Soxhlet extractor. Place the apparatus on a cold heating mantle and secure it in an up-right position.

10.6 Add Surrogate and Spike Solutions

- **NOTE:** The standards must be allowed to come to room temperature before spiking the samples.
- **NOTE:** Before adding surrogates and spikes, the extractionist must have their batch reviewed by a second person, and the review is documented on the Organic Extraction Checklists (see Work Instruction WI-DV-009).
- 10.6.1 Add the appropriate volume of the appropriate working surrogate standard to the sample in the Soxhlet extractor for each field sample and QC sample. Reference Work Instruction WI-DV-009 for the correct standard and the correct volume to add. Record the ID of the standard used on the benchsheet.
- 10.6.2 Add the appropriate volume of the appropriate working LCS and MS/MSD standard to the sample in the Soxhlet extractors containing any LCS, LCSD, MS, and MSD samples. Reference Work Instruction WI-DV-009 for the correct standard and the correct volume to add. Record the ID of the standard used on the benchsheet.
- **10.7** Attach a cold condenser to the top of the Soxhlet extractor body. Check to make sure that the condenser is cold and the hoses are not kinked.
 - **NOTE:** If the air is humid, a Friedrich's Condenser with a drying tube filled with Dryrite should be used to prevent water from condensing into the soxhlet.
- **10.8** Turn on the heating mantle to gently boil the solvent in the boiling flask. Check to make sure there are boiling chips in the flasks. Check the ground glass joints for leaks.

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- **10.9** Record the date and the time the extraction started on the benchsheet.
- 10.10 As the solvent evaporates from the boiling flask it will travel up the side arm to the condenser. The condensed solvent will then drip onto the soil sample, filling the extractor body until the sample is saturated. When the solvent in the extractor body reaches as high as the peak of the drain line, the solvent will start to siphon back into the boiling flask. Once all of the solvent has siphoned through, solvent will stop flowing through the drain line and will again start to collect in the extractor body.

NOTE: If the solvent does not stop flowing through the drain line, but rather continues to drip, then the extractor body will not become completely full again and only a portion of the soil sample will come in contact with the solvent dripping from the condenser. If this should happen, check to make sure that the extractor is in an upright position or slightly tipped so the drain line opening is elevated. Solvent may not flow through the drain line if the glass wool plug is too thick to allow the solvent to completely drain after the siphon started.

- **10.11** Continue the extraction for 18 to 24 hours.
- **10.12** After 18 to 24 hours, turn off the heating mantle and let the solvent in the boiling flask cool.
- **10.13** Record the date and the time the extraction stopped on the benchsheet.
- **10.14** If solvent remains in the Soxhlet extractor body, gently tilt the apparatus towards the drain line. This will cause the solvent to siphon one last time and collect in the boiling flask.
- **10.15** Remove the boiling flask, cap with aluminum foil, and store refrigerated at 0-6°C until it can be concentrated. If the extract contains visible solids it will be necessary to vacuum filter the extract prior to sending the extract on to concentration.
- **10.16** Dispose of the solid sample and the sodium sulfate into Waste Stream D.
- **10.17** Transfer into the LIMS any hand-written documentation and notes that were recorded on the benchsheet. This verification is documented on the Organic Extraction Checklist (see Work Instruction WI-DV-009).

11.0 Calibration

Not applicable to this procedure.

12.0 Calculations / Data Reduction

Not applicable to this procedure.

13.0 Method Performance

13.1 Method Detection Limit Study (MDL)

Before analyzing samples, the laboratory must establish a method detection limit (MDL). See DV-QA-005, "Determination of Method Detection Limits", for more information on the method detection limit studies.

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13.2 <u>Demonstration of Capabilities</u>

An initial demonstration of capability (IDOC) must be performed by each analyst. Ongoing proficiency must be demonstrated by each analyst on an annual basis. See DV-QA-0024, "Employee Training", for more information on the IDOCs.

13.3 Training Requirements

The group/team leader has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use and has the required experience. Further details concerning the training program are described in SOP# DV-QA-0024.

14.0 Pollution Control

The volume of spike solutions prepared is minimized to reduce the volume of expired standard solutions requiring hazardous waste disposal.

15.0 Waste Management

- 15.1 All waste will be disposed of in accordance with Federal, State, and local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this procedure, the policies in section 13, "Waste Management and Pollution Prevention", of the Corporate Environmental Health and Safety Manual, and DV-HS-001P, "Waste Management Program.
- **15.2** The following waste streams are generated when this method is carried out:
 - **15.2.1** Methylene chloride Waste Stream B
 - 15.2.2 Flammable solvent Waste Stream C
 - **15.2.3** 1:1 MeCl2:Acetone Waste Stream CA
 - **15.2.4** Solid waste/sodium sulfate Waste Stream D
 - **15.2.5** Expired Standards/Reagents Contact Waste Coordinator for guidance
- **15.3** Radioactive waste, mixed waste, and potentially radioactive waste must be segregated from non-radioactive waste as appropriate. Contact the Waste Coordinator for proper management of these materials.

16.0 References / Cross-References

- **16.1** Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition and all promulgated updates, January 2005.
 - **16.1.1** Method 3540C, Soxhlet Extraction, Revision 3, December 1996
 - **16.1.2** Method 3500C, Organic Extraction and Sample Preparation, Revision 3, February 2007.

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17.0 Method Modifications:

- **17.1.1** SW846 Method 3540C calls for extraction of 10 g of sample and 10 g of sodium sulfate. This SOP calls for the extraction of 30 g of sample and addition of enough sodium sulfate for the mixture to be free-flowing.
- **17.1.2** SW-846 Method 3540C calls for the use of 300 mL solvent in a 500 mL flask on the Soxhlet extractor. This SOP uses 250 mL solvent in a 250 mL flask.
- **17.1.3** SW846 Method 3540C calls for the extraction to last 16-24 hours. This SOP calls for the extraction to last 18-24 hours.

18.0 Attachments

Attachment 1: Determinative Methods Using Soxhlet Extraction

Attachment 2: Example Benchsheet

Attachment 3: Diagram of Soxhlet Extractor

19.0 Revision History

- Revision 5, dated 13 July 2012
 - Clarified Section 10.3.2 that the rinse can be performed with either methylene chloride or 1:1 methylene chloride: acetone.
 - Added Waste Stream CA to section 15.2
 - Added the note to Section 10.7 to give instructions on how to prevent condensation into the sample.
- Revision 4, dated 08 July 2011
 - Added section 1.4 to address percent moisture determination which is part of the source method but not part of this SOP
 - Added section 4.6 to address potential degradation of organophosphorus compounds
 - Revised Section 7 and Attachment 2 to remove details on the preparation of the surrogate and spike standards. Added instead a reference to DV-OP-0020 for the details of standard preparation.
 - Revised Attachment 2 to show an example benchsheet.
 - Source Method review
 - Updated method modifications
 - Added Method 3500C to references
 - Formatting and grammatical changes throughout
- Revision 3, dated 23 June 2010
 - TAL Denver implemented a new LIMS system since the last revision and therefore throughout the SOP references to "QC programs" and "SACs" were replaced with "Method Comments" and references to work instruction WI-DV-0032 were added.
 - The SOP was revised to include the use of cellulose thimbles.
 - The SOP was revised to include the use of hot plates.
 - Section 10.4 was revised to state that 30g to 33g of sample would be used instead of 30g +/- 2g.
 - Section 10.6 was revised to remove the reference to a witness for the addition of spikes and surrogates. Instead, the requirement for a reviewer was added.

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- Revision 2, dated 17 June 2009
 - Updated SOP references throughout SOP
 - Added the use of sodium sulfate to section 10.5.2
 - Added Note to section 10.10 concerning the use of a drying tube.
 - Added Imidan and Carbophenothion to Attachment 2.
 - Corrected Section 8 to say minimum sample size is 30g not 3g
 - Revised Section 8 to include the hold time for method 8141B
 - Removed Attachment "Organic Extraction Checklist" and referenced instead WI-DV-009.
 - Revised Attachment 2: Working Standards for Organophosphorus Pesticides to include additional compounds in the LCS standard.
- Revision 1, dated 25 April 2008
 - Integration for TestAmerica and STL operations.

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Attachment 1.

Determinative Methods Using Soxhlet Extraction

Method Description	Determinative Method	SOP
Organophosphorus Pesticides	SW846 8141A, 8141B, & EPA Method 614	DV-GC-0017

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Attachment 2. Example Benchsheet

Solid SW-846-3500 Analysis Sheet

(To Accompany Samples to Instruments)

 Batch Number: 280-73577
 Analyst: Guest, Erin E
 Batch Open: 6/23/2011
 3:12:00PM

Method Code: 280-3540C-280

Batch End: 6/28/2011 7:51:00PM

Soxhlet Extraction

	Input Sample Lab ID (Analytical Method)	SDG	Initial Amount	Final Amount	Due Date	Analytical TAT	Dlv Rank	Comments	Output Sample Lab ID
1	MB~280-73577/1 N/A	N/A	30.7 g	2000 uL	N/A	N/A	N/A		
2	LCS~280-73577/2 N/A	N/A	30.5 g	2000 uL	N/A	N/A	N/A		
3	280-17271-B-1 (8141A)	CUF1348	31.2 g	2000 uL	7/5/11	8_Days - R	2		
4	280-17271-A-2 (8141A)	CUF1348	31.4 g	2000 uL	7/5/11	8_Days - R	2		
5	280-17271-A-3 (8141A)	CUF1348	30.9 g	2000 uL	7/5/11	8_Days - R	2		
6	280-17300-D-2 (8141A)	N/A	30.3 g	2000 uL	7/5/11	7_Days - R	2		
7	280-17300-D-2~MS (8141A)	N/A	30.2 g	2000 uL	7/5/11	7_Days - R	2		
8	280-17300-D-2~MSD (8141A)	N/A	30.1 g	2000 uL	7/5/11	7_Days - R	2		2 8 0 - 1 7 3 0 0 - D - 2 - C M S D1
9	280-17300-F-4 (8141A)	N/A	30.5 g	2000 uL	7/5/11	7_Days - R	2		

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Attachment 2 Continued. Example Benchsheet

Solid SW-846-3500 Analysis Sheet

(To Accompany Samples to Instruments)

 Batch Number: 280-73577
 Analyst: Guest, Erin E
 Batch Open: 6/23/2011
 3:12:00PM

 Method Code: 280-3540C-280
 Batch End: 6/28/2011
 7:51:00PM

	Batch Notes
First End time	6/24/11 @ 1620
First Start time	6/23/11 @ 1530 (Heating mantles tripped overnight. Restarted @ 0645)
Perform Calculation (0=No, 1=Yes)	0
Nominal Amount Used	30
Prep Solvent Volume Used	250
Vendor of Reagent used	JT Baker
Person's name who did the	
	Reviewer: Cokley.C S/S: Guest.E pipette-F
reagent drop Na2SO4 Lot Number	
Silica Gel Lot Number	N/A
Concentration Start Time	N/A
Concentration End Time	N/A
Balance ID	24750402
Prep Solvent Name	1:1 Acetone/MeCl2
Prep Solvent Lot #	K18E56
Exchange Solvent Name	Hexane @ 50 mL
Exchange Solvent Lot #	K11E32
Blank Soil Lot Number	ZS00900
Florisil Lot #	N/A
TBA Lot #	N/A

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Batch Open: 6/23/2011 3:12:00PM

Attachment 2 Continued. Example Benchsheet

Analyst: Guest, Erin E

Batch Number: 280-73577

Solid SW-846-3500 Analysis Sheet

(To Accompany Samples to Instruments)

Comments			

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Attachment 2 Continued. Example Benchsheet

Solid SW-846-3500 Analysis Sheet

(To Accompany Samples to Instruments)

 Batch Number: 280-73577
 Analyst: Guest, Erin E
 Batch Open: 6/23/2011
 6/23/2011
 3:12:00PM

 Method Code: 280-3540C-280
 Batch End: 6/28/2011
 7:51:00PM

Reagent Additions Worksheet

Lab ID	Reagent Code	Amount Added	Final Amount	Ву	Witness
MB 280-73577/1	8141 Surr_00011	1 mL	2000 uL		
LCS 280-73577/2	8141 LCS_00018	1 mL	2000 uL		
LCS 280-73577/2	8141 Surr_00011	1 mL	2000 uL		
280-17271-B-1	8141 Surr_00011	1 mL	2000 uL		
280-17271-A-2	8141 Surr_00011	1 mL	2000 uL		
280-17271-A-3	8141 Surr_00011	1 mL	2000 uL		
280-17300-D-2	8141 Surr_00011	1 mL	2000 uL		
280-17300-D-2 MS	8141 LCS_00018	1 mL	2000 uL		
280-17300-D-2 MS	8141 Surr_00011	1 mL	2000 uL		
280-17300-D-2 MSD	8141 LCS_00018	1 mL	2000 uL		
280-17300-D-2 MSD	8141 Surr_00011	1 mL	2000 uL		
280-17300-F-4	8141 Surr_00011	1 mL	2000 uL		

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Attachment 2 Continued. Example Benchsheet

Solid SW-846-3500 Analysis Sheet

(To Accompany Samples to Instruments)

 Batch Number: 280-73577
 Analyst: Guest, Erin E
 Batch Open:
 6/23/2011
 3:12:00PM

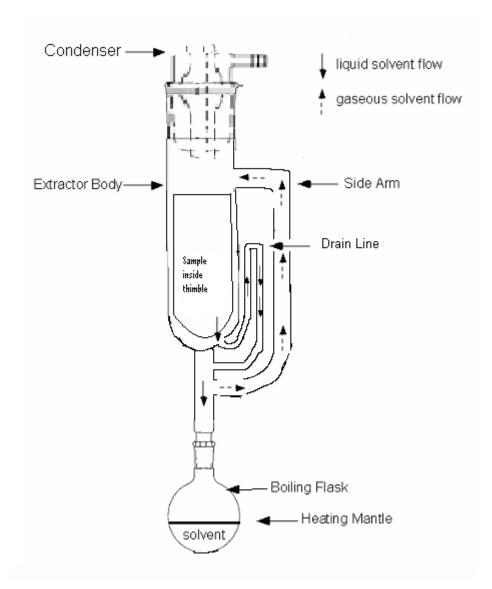
 Method Code: 280-3540C-280
 Batch End:
 6/28/2011
 7:51:00PM

Reagent	Amount/Units	Lot#:

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Attachment 3.

Diagram of Soxhlet Extractor





APPENDIX B Project Standard Operating Procedures



Standard Operating Procedure No. 1 Water Level Measurement

1.0 OBJECTIVE

The purpose of this document is to define the standard operating procedure (SOP) for measuring water elevations in monitoring wells included in environmental monitoring programs. This procedure describes equipment and field procedures necessary to collect water elevation measurements. The well locations and frequency of measurement are specified in project-specific work plans and QAPPs.

2.0 EQUIPMENT AND MATERIALS

The equipment and materials necessary that may be used to measure water levels include:

- Electronic water level indicator capable of producing measurements to a precision of 0.01 feet
- 5 gallon buckets or equivalent for decontamination
- Brushes for decontamination
- Water Level Measurement Form or Groundwater Sampling Form
- Field notebook
- Chemical-free paper towels or Kimwipes
- Alconox soap
- Potable water
- Garden-type spray bottle filled with deionized or distilled water
- Appropriate health and safety equipment

3.0 WATER ELEVATION MEASUREMENT PROCEDURE

3.1 DISCUSSION

Generally, water elevation measurements are used to construct potentiometric surface maps. Therefore, water level measurements at a given site should be collected within a 24 hour period. The device used to measure water levels should be adequate to attain an accuracy of 0.01 feet. Water levels should be allowed to stabilize for a minimum of 24 hours after well construction and development before measurements are taken.

3.2 MEASUREMENT PROCEDURE

This section gives the steps to follow when measuring water levels. Note that appropriate health and safety steps should be implemented and health and safety equipment should be worn during well opening, well measurement, and decontamination.

- Before any measurement is taken, the water level indicator shall be decontaminated. Decontamination procedures are discussed in SOP – Sampling Equipment Decontamination.
- Confirm that the monitoring well is labeled and the location ID is visible on the protective casing and that the ID coincides with the expected location.

- After opening the well cover, measure the depth of the static water level and the
 total depth of the well using an electronic water level indicator. The measuring
 point for all the wells shall be the top of PVC or steel well casing. The measuring
 point will be marked by a notch or other mark in the PVC or steel casing. If no mark
 is present, measure from the top of the north side of the casing.
- The static water level and the depth of the well shall be measured with the indicator, logged on the field data sheet or field notebook as feet below top of casing (ft.-TOC), and verified before the indicator is removed from the well. Note any significant changes in water level, by comparing the most recent measurement with past measurements, if appropriate.
- The water level depth below the measuring point (ft.-TOC) will be subtracted from the measuring point elevation to determine the elevation of the static water level. If measuring point elevations are available at the time of water level measurement, the calculated water elevation (ft.-MSL) should be checked in the field to see that it is reasonable and the subtraction was performed correctly. If there is a significant discrepancy in the measured water level or calculated water elevation, the well should be measured again.
- All columns of field data sheets shall be completed, including time of measurement.
 If items on the sheet do not apply to a specific location, the item will be labeled as not applicable (NA). A sample field data sheet for water elevation measurement is shown (Figure 1).

3.3 DECONTAMINATION

The water level indicator must be decontaminated before use, between wells, and at the conclusion of measurements. The probe will be decontaminated according to the procedure for decontamination of sampling equipment described in SOP – Sampling Equipment Decontamination.

4.0 DOCUMENTATION

Documentation of observations and data acquired in the field will provide information on the activities concluded and also provide a permanent record of field activities. The observations and data will be recorded with waterproof ink in a permanently bound weatherproof field logbook with consecutively numbered pages, and on field data sheets.

4.1 FIELD DATA SHEET FOR WATER LEVEL MEASUREMENTS

A field sampling data sheet for groundwater samples will be completed at each sampling location (sample attached). If items on the sheet do not apply to a specific location, the item will be labeled as not applicable (NA). The information on the data sheet includes the following:

- Well number
- Field book reference number

- Field personnel
- Well I.D.
- Date and time of measurements
- Sample identification number
- Water level (ft.-TOC)
- Static Water Elevation Data

The measurement point elevation (ft.-MSL) should be filled in if it has been determined at the time of measurement. If it is not known the form will be completed at a later time when further information is available. Any irregularities or problems that may have a bearing on sampling quality should be noted in the field.

Standard Operating Procedure No. 3 Sample Handling and Management

1.0 PURPOSE AND SCOPE

The purpose of this document is to define the standard operating procedure (SOP) for sample management including sample handling, documentation, and analysis for environmental samples collected for chemical analyses including: sediment, soil, surface water and groundwater. This procedure is intended to be used together with the other SOPs.

2.0 EQUIPMENT AND MATERIALS

The following equipment will be used for sample management:

- Shipping forms
- Sample containers
- Ziploc bags
- Ice
- Tape (clear and strapping)
- Scissors/knife
- Cooler/ice chest
- Custody seal
- Garbage bags
- Waterproof Pens
- Chain of Custody (COC) Forms
- Sample Labels
- Logbook
- Gloves
- Preservative (if necessary)
- Packing material
- Trip blank (as necessary)
- Temperature blank

3.0 PROCEDURES FOR SAMPLE HANDLING, DOCUMENTATION, AND ANALYSIS

3.1 SAMPLE LABELING

All sample labels should be filled out with waterproof ink. Soil and water sample labels may be supplied by the laboratory. For soil and sediment samples collected in jars and sample bottles for groundwater and surface water analyses, sample labels may be completed and attached prior to sample collection. Labels may be partially completed prior to sample collection. The date and time should not be completed until the time of sample collection. At a minimum, each label shall contain the following information:

- Project/Facility Name
- Grab or composite sample
- Sampler's company affiliation

- Date and time of sample collection
- Analyses required
- Preservation used
- Sampler's initials
- Filtered (if applicable)
- Sample identification (see Section 5.2 below)

3.2 SAMPLE NOMENCLATURE SCHEME

The sample identification (ID) varies significantly with each project. At minimum the sample ID should contain enough information to be correctly associated with a specific sampling location. The sample ID shall also be recorded on the sample form for the respective location. Additionally, Quality Control/Quality Assurance (QA/QC) samples should contain a sample ID such that the laboratory would not know it is a QA/QC sample.

3.3 SAMPLE HANDLING

This section discusses proper sample containers, preservatives, and handling and shipping procedures. The QAPP also summarize the information contained in this section and also includes the sample holding times for each analysis.

3.3.1 Sample Containers

Certified, commercially clean sample containers shall be obtained from the contract analytical lab. If appropriate, the bottles shall be labeled by the laboratory to indicate the type of sample to be collected. Required preservatives shall be prepared and placed in the bottles for aqueous analyses at the laboratory prior to shipment to the site.

3.3.2 Sample Preservation

With the exception of samples that are to be hand-delivered to the laboratory during the day of sample collection, samples will be stored on ice to obtain a temperature of 4°C in an insulated cooler immediately following sample collection. Samples delivered to the laboratory during the day of sample collection are acceptable if they have been placed on ice in an insulated cooler but have not yet reached a temperature of 4°C. Soil and sediment samples do not require additional preservation. As noted above, sample containers for aqueous samples will be obtained from the laboratory containing the appropriate preservatives.

3.4 SAMPLE SHIPPING

Sample containers will wrapped in protective packing material (if appropriate). Samples will then be placed in a cooler with ice for shipment to the laboratory. The drain on the cooler shall be taped shut. Samples collected in glass containers will be packed in foam liners and/or

bubble wrap to ensure that no breakage occurs during shipment. A temperature blank will be included in each cooler. Samples will be sent to the analytical laboratory via Federal Express or equivalent. Shipping receipts should be retained for documentation and sample tracking.

A completed chain-of-custody (COC) form for each cooler will be placed in a Ziploc bag and taped to the inside of the cooler lid. Coolers will be wrapped with packing tape at two locations to secure lids. Signed and dated custody seals shall be placed on the outside of each cooler in two places in such a manner as to allow detection of tampering (e.g., the seals must be broken to open the cooler).

3.5 HOLDING TIME REQUIREMENTS

The holding time is specified as the maximum allowable time between sample collection and analysis and/or extraction, based on the analyte of interest, stability factors, and preservation methods. Allowable holding times for chemical analysis parameters are listed in the QAPP. Samples should be sent to the laboratory after collection in sufficient time to allow the laboratory to meet holding time requirements.

4.0 QUALITY CONTROL REQUIREMENTS

QC requirements relevant to analysis of environmental samples shall be followed during analytical activities to meet the quality objectives and criteria. The purpose of the QC program is to produce data of known and documented quality that satisfy the project objectives and that meet or exceed the requirements of the standard methods of analysis.

4.1 QC SAMPLES

A number of QC samples will be employed to assess various data quality parameters, such as representativeness of the environmental samples, the precision of sample collection and handling procedures, the thoroughness of the field equipment decontamination procedures, and the accuracy of laboratory analysis. Types of QC samples are discussed below.

4.1.1 Matrix Spike/Matrix Spike Duplicate

Matrix spike (MS) and matrix spike duplicate (MSD) samples are prepared by spiking additional aliquots of sample with known concentrations of all project target analytes.

The sample to be used for the MS/MSD analyses shall be designated on the chain of custody and additional sample volume shall be submitted, as necessary. The MS/MSD results are used to document the bias of a method due to sample matrix. Consequently, MSs and MSDs are not used to control the analytical process. Minimum numbers of MS and one MSD samples are indicated in the project-specific QAPP, generally one for every 20 environmental samples of a given matrix. Alternately, a laboratory may prepare and analyze a MS sample and a laboratory duplicate sample as discussed below. Analysis of a MS/MSD or MS/LD sample set to assess matrix effects on accuracy and precision is typically dependent on the analyte class (e.g., inorganic vs. organic) and the likelihood of detecting the target analyte.

4.1.2 Rinsate Blank

A rinsate blank is a sample of ASTM Type II reagent grade water poured into or over or pumped through the sampling device, collected in a sample container, and transported to the laboratory for analysis. ASTM Type II reagent grade water obtained from the laboratory is used to prepare the rinsate blank sample. Rinsate blanks are used to assess the effectiveness of equipment decontamination procedures used to prevent cross-contamination between sampling locations. The frequency of collection for rinsate blanks is indicated in the project-specific QAPP, generally a minimum of 1 rinsate blank for every 20 environmental samples collected with a given type of sampling equipment, and only for sampling equipment which is decontaminated and reused to collect environmental samples. Rinsate blanks will be prepared in a manner identical to samples and shall be analyzed for all laboratory analyses requested for the environmental samples collected at the site using the subject equipment. Rinsate blanks are not necessary for disposable or dedicated sampling equipment.

4.1.3 Trip Blank

The trip blank consists of a VOC sample vial filled in the laboratory with ASTM Type II reagent grade water, transported to the sampling site, handled in the same manner as an environmental sample and returned to the laboratory for analysis. Trip blanks are not opened in the field. Trip blanks are prepared only when VOC samples are taken and are analyzed only for VOC analytes. Trip blanks are used to assess the potential introduction of contaminants from sample containers or during the transportation and storage procedures. One trip blank shall accompany each cooler containing samples for VOC analysis that is sent to the laboratory.

4.1.4 Field Duplicates

A field duplicate sample is a second, discrete sample volume collected at the same location as the original sample (homogenization is not performed between the original sample and the field duplicate). Aqueous field duplicate samples are collected from successive volumes from the same sample source and device (e.g., bailers). Sediment and soil field duplicates are collected in succession from the same sample source and device. Individual analytes for the primary and duplicate groundwater samples are to be collected in order (e.g. VOC primary then VOC duplicate, etc.) so that a long period of time does not pass between collection of each analyte. Field duplicate samples are collected using identical recovery techniques, and treated in an identical manner during storage, transportation, and analysis. The sample containers are assigned an identification number in the field such that they cannot be identified (blind duplicate) as field duplicate samples by laboratory personnel performing the analysis.

Field duplicate sample results are used to assess precision of the sample collection process and the heterogeneity of the medium sampled. The frequency of collection for field duplicates is indicated in the project-specific QAPP, generally a minimum of one field duplicate sample from each group of 20 environmental samples of a given matrix. Specific locations for collection of field duplicate samples may be designated prior to the beginning of sample collection.

5.0 DOCUMENTATION AND TRACKING

5.1 FIELD NOTES

Documentation of observations and data acquired in the field will provide information on the acquisition of samples and also provide a permanent record of field activities. The observations and data will be recorded with waterproof ink in a permanently bound weatherproof field logbook with consecutively numbered pages and, if applicable, on field sampling data sheets.

The information in the field logbook will include the following as a minimum. Unless information is recorded on a field sample collection form and that form is cross referenced in the logbook entry. Additional information is included in the specific SOPs regarding the appropriate data sheets.

- Project name
- Location of sample
- Sampler's signature
- Date and time of sample collection
- Sample identification numbers and sample depth (if applicable)
- Description of samples (matrix sampled), composite or grab sample
- Description of QA/QC samples (if collected)
- Sample methods or reference to the appropriate SOP
- Field observations
- Decontamination information
- Calibration information
- Personnel present
- Method of shipment
- Any deviations from SOPs
- Any information pertaining to the sample that is not noted of the sample form.

If samples are held for an extended period of time (i.e., inadvertently missed Fed-Ex pick up), field personnel will document all sample handling and custody in the field logbook.

5.2 CHAIN-OF-CUSTODY FORM

A record of each sample collected will be indicated on a COC form. Every sample in the coolers shall be covered by the COC form(s) accompanying the coolers. Coolers may contain a single COC covering only the samples in that cooler or may contain copies of the COC that covers all of the samples in all of the coolers. One cooler must contain the original COC. The COC form will provide an accurate written record which can be used to trace the custody of all samples from the time of collection through data analyses and reporting.

The following will be specified for each sample on the COC form as a minimum:

- Sample ID
- Sample date

- Sample time
- Requested analysis
- Number of containers
- Sampler's signature or initials
- Preservation technique
- Sample type (i.e., medium)

Also recorded on the COC is the signature of the person relinquishing custody, the date and time that custody was relinquished, the name and address of the laboratory, and the name and phone number of a contact person regarding the shipment.

A sample is considered in custody if it is:

- 1. In one's actual possession
- 2. In view, after being in physical possession
- 3. Locked so that no one can tamper with it, after having been in physical custody
- 4. In a secured area

The person responsible for custody of the sample prior to delivery of the samples to the laboratory will sign the COC form, retain the last copy of the three-part COC form, document the method of shipment, and send the original and the second copy of the COC form with the sample (taped in a Ziploc bag to inner cooler lid). Upon receipt at the laboratory, the person receiving the samples will sign the COC form and return the second copy to the Project Manager or Quality Assurance Manager or specified designee. Copies of the COC forms and all custody documentation will be received and kept in the central files. The original COC forms will remain with the samples until final disposition of the samples by the laboratory. The analytical laboratory may dispose of the samples in an appropriate manner 60 to 90 days after data reporting. After sample disposal, a copy of the original COC will be sent to the Project Manager or Quality Assurance Manager or specified designee by the analytical laboratory to be incorporated into the central files.

SOP No. 4 – Sampling Equipment Decontamination

Standard Operating Procedure No. 4 Sampling Equipment Decontamination

SOP No. 4 – Sampling Equipment Decontamination

1.0 OBJECTIVE

Decontamination is performed as a quality assurance measure and safety precaution. It helps prevent cross-contamination among samples and helps maintain a clean working environment for the safety of field personnel.

2.0 EQUIPMENT AND MATERIALS

- Cleaning liquids such as soap or detergent solutions (Alconox or equivalent), potable water and distilled water
- Cleaning brushes
- · Cleaning containers, such as plastic buckets or tubs
- Pump sprayers for dispensing rinse waters
- A high-pressure hot water sprayer for cleaning large equipment (e.g., drill rods)
- Waste containers
- Health and safety equipment as outlined in the Site-Specific Health and Safety Plan

3.0 METHODOLOGY

Small, reusable equipment is decontaminated primarily by rinsing with liquids that include soap or detergent solutions, potable water, and distilled water. Steam cleaning may he used whenever visible contamination exists on large machinery or vehicles. Following decontamination, if the equipment is not to be reused immediately, it should be stored and protected from recontamination.

3.1 PRE-SAMPLING DECONTAMINATION ACTIVITIES

- Don the appropriate personal protective equipment, including nitrile gloves, as specified in the Site-Specific Health and Safety Plan and as required for the specific work area.
- 2. Assemble containers and equipment for decontamination.
- 3. Decontaminate new equipment or equipment not previously decontaminated before use. Disposable equipment, including polyethylene tubing and bailers, do not require decontamination prior to use.
- 4. Rinse equipment not previously decontaminated and appropriately protect from recontamination before the next use.

3.2 DECONTAMINATING SAMPLING EQUIPMENT

- 1. Remove solid particles from the equipment or material by brushing and rinsing with potable water. This will remove gross contamination.
- 2. Wash equipment with a brush and a phosphate-free detergent solution (Alconox or similar laboratory detergent).
- 3. Rinse equipment thoroughly with potable water.
- 4. Triple rinse the equipment with distilled water.

SOP No. 4 – Sampling Equipment Decontamination

5. Unless the equipment is going to be used immediately protect it from recontamination before the next use.

3.3 DECONTAMINATING LARGE EQUIPMENT

Drilling equipment (rigs, drill rods, augers, rods, bits, casing, screen. etc.), downhole logging equipment, and other large pieces of field equipment may be high-pressure steam-cleaned before and after use. Steam cleaning will be performed at an appropriate decontamination area specified by the field supervisor. The decontamination area shall be capable of containing decontamination fluids and solids. The decontamination fluids shall be managed in accordance with SOP – IDW Management.

Additionally, the drilling subcontractor has the responsibility of making the drilling rig free of leaks (i.e., hydraulic fluid, oil, gas. etc.) that could contaminate the boreholes. Grease may be sparingly used on rod shoulders to ease rod breaking upon completion of a borehole. Rod joints should be wiped with a clean cloth to minimize the amount of grease on the exterior of the rod.

4.0 COMMENTS

Decontamination is critical for maintaining the integrity of the sampling program. Check equipment carefully prior to sampling, and if there is any doubt about the effectiveness of the decontamination, repeat the decontamination process as an extra precaution. Decontamination fluids will be containerized and disposed of following the procedures provided SOP – IDW Management. Decontamination procedures shall be documented in the field log book.

SOP No. 5 – Soil Sampling for Chemical Analysis

Standard Operating Procedure No. 5 Soil Sampling for Chemical Analysis

SOP No. 5 – Soil Sampling for Chemical Analysis

1.0 OBJECTIVE

Soil samples will be collected for field screening and chemical analysis to help characterize the source areas and to determine the nature and extent of contamination in soil. Soil samples will be collected during direct push activities and monitoring well drilling.

2.0 EQUIPMENT AND MATERIALS

- Appropriate number and types of sample containers
- Precleaned stainless steel sampling utensils (See SOP Sampling Equipment Decontamination)
- Sample coolers and ice.
- Appropriate field documentation forms and labels and an indelible ink pen.
- Sampling equipment (split--spoons samplers).
- Decontamination equipment.
- Waste containers.
- Health and safety equipment, as specified in the Health and Safety Plan.

3.0 METHODOLOGY

Soil samples collected by direct push or split-spoon methods will be collected as follows (See SOP – Direct Push Drilling and SOP – Hollow Stem Auger Drilling):

- a) Retrieve sampler from borehole and remove drive shoe and head assembly. Open the sampler carefully to avoid disturbing the sample.
- b) Once the sampler has been opened, the length of the sample will be screened with a photoionization detector (PID), and the reading will be recorded on the drilling log.
- c) The upper portion of soil in the sampler potentially represents material that has fallen from above or has been scraped from the sides of the auger hole; therefore, this portion is not representative of the sampling interval and should be discarded.
- d) Samples for volatile organic compound (VOC) analysis will be collected first to minimize the potential for volatilization. A sufficient amount of soil will be collected and transferred directly to VOC sample containers using a stainless steel utensil. The sample will be packed to completely fill the container and reduce the amount of headspace, which will minimize the loss of volatile compounds. Teflon-lined septum lids will be immediately secured on the sample containers.
- e) Collect samples for additional analysis next.
- f) Complete the sample labels in accordance with SOP Sample Handling and Management and place them on the jars.
- g) Place protective packing on the sample jar.

SOP No. 5 - Soil Sampling for Chemical Analysis

- h) Decontaminate sampling equipment in accordance with SOP Sampling Equipment Decontamination.
- i) Collect and manage wastes as specified the waste management plan.

4.0 COMMENTS

Following standard practice, the soil samples for VOC analysis will be collected before any logging or other sample handling is conducted. Due to the nature of VOCs, it is critical to collect these samples as quickly as possible and to immediately place them in a cooler with ice. The sample jars for VOC soil samples will be pre-chilled in a cooler with ice prior to filling the jars with soil. This practice will further reduce the potential for volatilization during sample collection. It may be necessary to advance an additional boring for the purpose of lithologic logging.

4.1 LITHOLOGIC LOGGING AND GEOTECHNICAL ANALYSIS

Lithologic logging will be performed to define the subsurface geology. Selected samples will be collected to send to the laboratory for quantitative geotechnical analysis. See SOP – Borehole Logging for further information.

SOP No. 6 - IDW Management

Standard Operating Procedure No. 6 IDW Management

SOP No. 6 – IDW Management

1.0 OBJECTIVE

This Standard Operating Procedure (SOP) provides technical guidance and methods that will be used for the handling, management, and disposal of investigation derived waste (IDW) encountered or generated during environmental investigation activities. This SOP gives descriptions of equipment, field development procedures, field data collection, and personnel responsibilities.

2.0 EQUIPMENT AND MATERIALS

The following equipment and materials may be needed for IDW management:

Personal protective equipment (PPE) as outlined in the HSP

- Decontamination equipment and supplies (e.g., wash/rinse tubs, brushes, alconox, plastic sheeting, paper towels, sponges, baby wipes, garden-type water sprayers, large plastic bags (minimum 0.55 mil), potable water, distilled water and/or deionized water)
- Department of Transportation (DOT)-rated 55-gallon drums or other approved containers for containing soil cuttings, decontamination water, and formation water
- Drum/bung wrench and drum funnel
- Heavy equipment forklift or vehicle with drum grappler (as necessary)
- Laboratory-supplied sample containers
- Photoionization detector (PID) or flame ionization detector (FID)
- Wood pallets (as necessary)
- Non-porous (e.g., stainless steel) shovels
- Polyethylene tanks (as necessary)
- Field notebook and waterproof and permanent marking pens

3.0 PROCEDURES

It is anticipated that both non-liquid and liquid IDW will be generated or encountered during field activities. IDW generated during the field investigation is expected to include:

- Soil cuttings and other soil wastes generated during sampling
- Well development and purged water
- Wash and rinse waste from decontamination activities
- Used PPE and other non-soil solid wastes

Sections 5.1 and 5.2 describe procedures for disposal of IDW on-site. Section 5.4 addresses management and disposal requirements for off-site disposal and potentially hazardous materials.

3.1 SOIL IDW

• Soil cuttings generated during drilling and soil sampling will be placed into DOT-rated 55-gallon drums, or appropriately sized containers at the point of generation.

SOP No. 6 – IDW Management

- Mixing of the cuttings from several borings or sampling locations is permissible in order to fill the drums.
- When drums or containers are full, or daily activities are completed, the drum lids and rings will be fastened. Full drums or containers will be transported to the designated IDW accumulation area on a regular basis to avoid accumulation of drums or containers at individual investigation sites for extended periods of time.
- Drums will be stored on pallets at the designated IDW accumulation area. Drums will be segregated to separate soil from liquid IDW.
- Drums will be sealed and labeled with permanent markings (using paint pens or drum labels) with the following information:
 - Source: the boring(s), well, or site identification number
 - Matrix (e.g., soil, water)
 - Sample interval
 - o Fill date
 - o Drum identification number
 - o Contractor
 - o Point of contact with phone number
 - Labeled "Contents Pending Analysis"
- If large volumes of soil IDW will be generated, soil IDW will be transferred from the drums into roll-off bins (lined and covered) located within the designated IDW accumulation area.
- If no associated investigation sample results exist, a composite soil sample will be collected from the soil IDW drums by collecting a drive or hand auger sample from each of the drums. The sample material from all of the drums will be composited into a single sample that will be used to characterize and dispose of the IDW.

3.2 LIQUID IDW

- Well development, purge, abandonment, and decontamination water will be contained in DOT-rated drums, or in appropriately sized watertight containers, at the point of generation.
- When drums or tanks are full, or daily activities are completed, the containers will be sealed: for example, drum lids and rings will he fastened.
- Drums will be stored on pallets at the designated IDW accumulation area. Drums will be segregated to separate soil from liquid IDW.
- Drums will be sealed and labeled with permanent markings (using paint pens or drum labels) with the following information:
 - Source: the boring(s), well, or site identification number
 - Matrix (e.g., soil, water)
 - Sample interval
 - Fill date
 - o Drum identification number
 - Contractor
 - Point of contact with phone number

SOP No. 6 – IDW Management

- Labeled "Contents Pending Analysis"
- If large volumes of water will be generated, the water will be transferred into an appropriately-sized polyethylene tank.
- If no associated investigation sample results exist, a composite water sample will be collected from the water IDW drums. The sample will be used to characterize and dispose of the IDW.

3.3 PPE AND DISPOSABLE INVESTIGATION EQUIPMENT

- The plan for managing used PPE and other non-soil solid waste generated during field activities (e.g., sample handling) will be segregated separately and placed into dedicated heavy duty plastic bags or containers (e.g. drums)
- Potentially contaminated PPE or disposable investigation equipment will be decontaminated prior to placement in the plastic bags or containers, if warranted.
- Decontamination procedures consist of brushing off, or using small amounts of water to scrub off, gross potential contamination.

3.4 DISPOSAL OF IDW

IDW will be disposed of in accordance with federal, state, and local regulations.

4.0 DOCUMENTATION

Project staff are responsible for thoroughly documenting IDW handling and disposal activities. Personnel will be responsible for documenting the collection, transportation, labeling (if applicable), and staging or disposition of IDW. A Waste Inventory Tracking Form is to be completed if necessary. Documentation should include the following:

- Project Name
- Names of personnel
- Site location
- Type of activities
- Date waste generated
- Boring, well, or site number(s)
- Matrix
- Type of container(s)
- Estimated volume
- Disposition of contents
- Comments (field evidence of contamination (e.g., PID reading, odors)
- Any variance to procedures described in this SOP

SOP No. 7 - Lithologic Logging

Standard Operating Procedure No. 7 Lithologic Logging

SOP No. 7 – Lithologic Logging

1.0 OBJECTIVE

Lithologic logging will be performed to define the subsurface geology. Selected samples will be collected to send to the laboratory for quantitative geotechnical analysis. Soils will be described using the Unified Soils Classification System (American Society for Testing and Materials [ASTM] Designation D 2488-09a: Standard Practice for Description and Identification of Soils [Visual-Manual Procedure]). This SOP serves as a supplement to the site-wide and investigation area specific workplans and field sampling plans (FSPs), and is intended to be used in conjunction with the other SOPs.

2.0 EQUIPMENT AND MATERIALS

The following materials and equipment may be needed for soil sampling using hand auger techniques:

- Munsell soil color chart
- Hand lens
- Putty knife or spatula
- Dropper with 10% hydrochloric acid (HCI) for calcium carbonate test
- Drilling forms
- Sampling device (e.g., core barrel, split-spoon, macrocore)
- Waste containers as specified in Appendix D
- Health and safety equipment, as specified in the Site-Specific Health and Safety Plan

3.0 PROCEDURES

A "site geologist" (geologist, hydrogeologist or geotechnical engineer) experienced in borehole drilling and soil sampling will be present at each operating drill rig. This site geologist will be responsible for logging samples, monitoring drilling operations, and preparing field boring logs.

- Boring log information will be recorded on a Boring Log Form. Depth information will be recorded to
 the nearest 0.1 foot. An appropriate scale will be used on the boring log form (e.g., a scale of 1 inch on
 the log form equaling 1 foot of boring).
- Measure entire sample core length and record the recovery on the drilling log. Mark lithologic changes on the drilling form. Lithologies will be logged directly from cores/samples and indirectly interpolated using professional judgment, drill cuttings, drill action, etc., between sampling intervals.
- Descriptions of intact unconsolidated soil samples will include parameters listed in Table 1. Material will be described in the following order:
 - Material type (i.e., sand [sandstone], silt [siltstone], clay [claystone], etc.)
 - Color (Munsell color chart will be used)
 - ➤ Grain size, sorting, rounding, and composition of the material (for sand or gravel)
 - > Types and amounts of secondary constituents
 - Other pertinent characteristics (plasticity, hardness, bedding, etc.)
 - Moisture content
 - USCS code (for unconsolidated material)

SOP No. 7 - Lithologic Logging

- Unconsolidated materials will be classified in accordance with the USCS (equivalent to ASTM D 2488-09a, "Description and Identification of Soil [Visual Manual Procedure]"; Attachment B and USEPA Manual 625/12-91/002 "Description and Sampling of Contaminated Soils"). Soil classifications will be made in the field at the time of sampling by the site geologist (Table 1).
- In the field, visual estimates of the volume of secondary soil constituents will be reported by such terms as "trace" (<5 percent), "few" (5-10 percent), "little" (15-25 percent) "some30-45 percent), and "mostly" (50-100 percent) or by an estimated percentage.
- Consolidated material (e.g., igneous and metamorphic rocks) will be described by parameters listed in Table 2 and described in Tennisen (1983), ASTM D5434-97, "Standard Guide for Field Logging of Subsurface Explorations of Soil and Rock", and ASTM C294-86(1991), "Standard Descriptive Nomenclature for Constituents of Natural Mineral Aggregates". Material will be logged using drill cuttings and/or rock core. Material will be described in the following order:
 - Rock Type
 - Color (Munsell color chart will be used)
 - Grain size and shape
 - Texture (stratification. foliation)
 - Mineral composition
 - Weathering and alteration
 - Strength
 - Other relevant notes
- Drill cuttings will be described in terms of the appropriate parameters, to the extent practical.
 "Classification" will be minimally described for this material, along with a description of drilling actions for the corresponding depth. Notations will be made on the log that these descriptions are based on observations of material other than formal samples (e.g., from cuttings).
- The drilling equipment used will be described on each log. Information such as drill rod size, bit size and type, and rig manufacturer and model will be recorded.
- All special problems encountered during drilling and their resolution will be recorded on the log. This
 would include sudden tool drops, unrecovered tools in the borehole, and lost casing.
- The dates for the start and completion of borings will be recorded on the log.
- Stratigraphic/lithologic changes will be identified on the boring log with a solid line at the measured borehole depths at which changes occur. Gradational transitions and changes identified from cuttings or methods other than direct observation and measurement will be identified by a horizontal dashed line at the appropriate scale depth based on the best judgment of the logger.
- Logs will show borehole and sample diameters and depths at which drilling or sampling methods or equipment change.
- Logs will show total depth of penetration and sampling. The bottom of the hole will be clearly identified on the log.
- Logs will identify the depth at which water is first encountered, the depth of water at the completion of drilling, and the stabilized depth to water. The absence of water in borings will also be indicated.
 Stabilized water-level data will include time allowed for levels to stabilize.
- Logs will include other information relevant to a particular investigation, but not limited to:
 - Odors
 - Field screening or test results (e.g., organic vapors)

SOP No. 7 – Lithologic Logging

- Any observed evidence of contamination in samples and cuttings
- Special abbreviations used on a log will be defined either in the log where used, or in a general legend.

6.0 DOCUMENTATION

Project staff are responsible for documenting sampling activities. A field boring log form will be completed summarizing field activities. Field notes will also be kept during drilling and logging activities. The following information will be recorded in a bound field log book:

- Names of personnel
- Weather conditions
- Date and time of drilling and sampling
- Location and sample station number
- Times that procedures and measurements are completed
- Decontamination times
- Calibration information
- Boring log information
- Other applicable information

3.2 DECONTAMINATION

All tools and sampling equipment (e.g., spatula, drive sampler, etc.) will be decontaminated between sample locations and between individual samples. Decontamination will be conducted in accordance with SOP – Sampling Equipment Decontamination.

4.0 DOCUMENTATION

Project staff are responsible for documenting sampling activities. A field boring log form will be completed summarizing field activities. Field notes will also be kept during drilling and logging activities. The following information will be recorded in a bound field log book:

- Names of personnel
- Weather conditions
- Date and time of drilling and sampling
- Location and sample station number
- Times that procedures and measurements are completed
- Decontamination times
- Calibration information
- Boring log information
- Other applicable information

SOP No. 7 - Lithologic Logging

TABLE 1 DESCRIPTION OF UNCONSOLIDATED SOIL

Parameter	Example
Formation, (if named and if known)	Alluvium
Unified Soil Classification System	Sandy Clay
Secondary Components and Estimated Quantities either by percentages or by descriptive percentage ranges (Note: terms used to indicate ranges should be described on the log or in a general legend)	sand: fine, with trace of med.
Color	gray
Consistency (cohesive soil). Use relative term	very soft, soft, medium, stiff, very stiff, hard
Density (non-cohesive soil). Use relative term	loose, medium, dense, very dense
Moisture Content. (Use relative term. Do not express as a percentage unless a value has been measured)	dry, damp, moist, wet, saturated
Texture/Fabric/Bedding	no apparent bedding, numerous vertical iron-stained tight fractures
Grain Angularity	rounded sand grains
Sorting (sands)	poorly sorted
Structure	slickensides
Grain or fragment size	coarse
Note "Fill", "Top of Natural Ground", and "Top of Bedrock" where appropriate	





FCC Information

Contains FCC ID: PI4411B

The enclosed device complies with part 15 of the FCC rules. Operation is subject to the following conditions: (1) This device may not cause harmful interference, and (2) This device must accept any interference received, including interference that may cause undesired operation.

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Read Before Operating

This manual must be carefully read by all individuals who have or will have the responsibility of using, maintaining, or servicing this product. The product will perform as designed only if it is used, maintained, and serviced in accordance with the manufacturer's instructions. The user should understand how to set the correct parameters and interpret the obtained results.

CAUTION!

To reduce the risk of electric shock, turn the power off before removing the instrument cover. Disconnect the battery before removing sensor module for service. Never operate the instrument when the cover is removed. Remove instrument cover and sensor module only in an area known to be non-hazardous.

Special Notes



When the instrument is taken out of the transport case and turned on for the first time, there may be some residual organic or inorganic vapor trapped inside the detector chamber. The initial PID sensor reading may indicate a few ppm. Enter an area known to be free of any organic vapor and turn on the instrument. After running for several minutes, the residual vapor in the detector chamber will be cleared and the reading should return to zero.



The battery of the instrument discharges slowly even if it is turned off. If the instrument has not been charged for 5 to 7 days, the battery voltage will be low. Therefore, it is a good practice to always charge the instrument before using it. It is also recommended to fully charge the instrument for *at least 10 hours* before first use. Refer to this User Guide's section on battery charging for more information on battery charging and replacement.

WARNINGS

STATIC HAZARD: Clean only with damp cloth.

For safety reasons, this equipment must be operated and serviced by qualified personnel only. Read and understand instruction manual completely before operating or servicing.

Use only RAE Systems battery packs, part numbers 059-3051-000, 059-3052-000, and 059-3054-000. This instrument has not been tested in an explosive gas/air atmosphere having an oxygen concentration greater than 21%. Substitution of components may impair intrinsic safety. Recharge batteries only in non-hazardous locations.

Do not mix old and new batteries or batteries from different manufacturers.

The calibration of all newly purchased RAE Systems instruments should be tested by exposing the sensor(s) to known concentration calibration gas before the instrument is put into service.

For maximum safety, the accuracy of the instrument should be checked by exposing it to a known concentration calibration gas before each day's use.

Do not use USB/PC communication in hazardous locations.

AVERTISSEMENT

DANGER RISQUE D'ORIGINE ELECTROSTATIQUE: Nettoyer uniquement avec un chiffon humide.

Pour des raisons de sécurité, cet équipment doit être utilisé, entretenu et réparé uniquement par un personnel qualifié. Étudier le manuel d'instructions en entier avant d'utiliser, d'entretenir ou de réparer l'équipement.

Utiliser seulement l'ensemble de batterie RAE Systems, la reference 059-3051-000 au 059-3052-000 au 059-3054-000. Cet instrument n'a pas été essayé dans une atmosphère de gaz/air explosive ayant une concentration d'oxygène plus élevée que 21%. La substitution de composants peut compromettre la sécurité intrinsique. Ne charger les batteries que dans emplacements désignés non-dangereuse.

Ne pas melanger les anciennes et les nouvelles batteries, ou bien encore les batteries de differents fabriquants.

La calibration de toute instruments de RAE Systems doivent être testé en exposant l'instrument a une concentration de gaz connue par une procédure diétalonnage avant de mettre en service l'instrument pour la première fois.

Pour une securite maximale, la sensibilité du l'instrument doit être verifier en exposant l'instrument a une concentration de gaz connue par une procédure diétalonnage avant chaque utilisation journalière.

Ne pas utiliser de connection USB/PC en zone dangereuse.

Standard Contents

Instrument
Calibration Kit
Charging Cradle
AC/DC Adapter
Alkaline Battery Adapter
Data Cable
CD-ROM With User's Guide, Quick Start Guide, and related materials

General Information

The compact instrument is designed as a broadband VOC gas monitor and datalogger for work in hazardous environments. It monitors Volatile Organic Compounds (VOC) using a photoionization detector (PID) with a 9.8 eV, 10.6 eV, or 11.7 eV gas-discharge lamp. Features are:

Lightweight and Compact

- Compact, lightweight, rugged design
- Built-in sample draw pump

Dependable and Accurate

- Up to 16 hours of continuous monitoring with rechargeable battery pack
- Designed to continuously monitor VOC vapor at parts-permillion (ppm) levels

User-friendly

- Preset alarm thresholds for STEL, TWA, low- and high-level peak values.
- Audio buzzer and flashing LED display are activated when the limits are exceeded.

Datalogging Capabilities

• 260,000-point datalogging storage capacity for data download to PC

The instrument consists of a PID with associated microcomputer and electronic circuit. The unit is housed in a rugged case with a backlit LCD and 3 keys to provide easy user interface. It also has a built-in flashlight for operational ease in dark locations.

Physical Description

The main components of the portable VOC monitoring instrument include:

- Three keys for user to interact with the instrument: 3 operation/programming keys for normal operation or programming
- LCD display with back light for direct readout and calculated measurements
- Built-in flashlight for illuminating testing points in dark environments
- Buzzer and red LEDs for alarm signaling whenever exposures exceed preset limits
- Charge contacts for plugging directly to its charging station
- Gas entry and exit ports
- USB communication port for PC interface
- Protective rubber cover

Specifications

Size: 9.25" L x 3.6" W x 2.9" H

Weight: 28 oz with battery pack

Detector: Photoionization sensor with 9.8, 10.6, or

11.7 eV UV lamp

Battery: A 3.7V rechargeable Lithium-Ion battery pack

(snap in, field replaceable, at non-hazardous

location only)

Alkaline battery holder (for 4 AA batteries)

Battery Charging: Less than 8 hours to full charge

Operating Hours: Up to 16 hours continuous operation

Display: Large dot matrix screen with backlight

Measurement range & resolution

Lamp	Range	Resolution
10.6 eV	0.1 ppm to 15,000 ppm	0.1 ppm
9.8 eV	0.1 ppm to 5,000 ppm	0.1 ppm
11.7 eV	0.1 ppm to 2,000 ppm	0.1 ppm

Response time (T_{90}) : 2 seconds

Accuracy 10 to 2000 ppm: $\pm 3\%$ at calibration point.

(Isobutylene):

PID Detector: Easy access to lamp and sensor for cleaning

and replacement

Correction Factors: Over 200 VOC gases built in (based on RAE

Systems Technical Note TN-106)

Calibration: Two-point field calibration of zero and

standard reference gases

Calibration Reference: Store up to 8 sets of calibration data, alarm

limits and span values

Inlet Probe: Flexible 5" tubing

Radio module: Bluetooth (2.4GHz), RF module (433MHz,

868MHz, 915MHz, or 2.4GHz)

Keypad: 1 operation key and 2 programming keys; 1

flashlight switch

Direct Readout: Instantaneous, average, STEL, TWA and peak

value, and battery voltage

Intrinsic Safety: US and Canada: Class I, Division 1, Groups A,

B, C, D

Europe: ATEX (0575 Ex II 2G Ex ia

IIC/IIB T4 Gb)

KEMA 07 ATEX 0127

Complies with EN60079-0:2009,

EN60079-11:2007

IECEx CSA 10.0005 Ex ia IIC/IIB T4 Gb

Complies with IEC 60079-0:2007,

IEC 60079-11:2006

(IIC: 059-3051-000 Li-ion bat pack or 059-3054-000 NiMH bat pack; IIB: 059-3052-000 alkaline bat pack)

EM Interference: Highly resistant to EMI/RFI. Compliant with

EMC R&TTE (RF Modules)

Alarm Setting: Separate alarm limit settings for Low, High,

STEL and TWA alarm

Operating Mode: Hygiene or Search mode

Alarm: Buzzer 95dB at 30cm and flashing red LEDs

to indicate exceeded preset limits, low battery

voltage, or sensor failure

Alarm Type: Latching or automatic reset

Real-time Clock: Automatic date and time stamps on datalogged

information

Datalogging: 260,000 points with time stamp, serial number,

user ID, site ID, etc.

Communication: Upload data to PC and download instrument

setup from PC via USB on charging station.

Sampling Pump: Internally integrated. Flow rate: 450 to 550

cc/min.

Temperature: -20° C to 50° C (-4° to 122° F)

Humidity: 0% to 95% relative humidity (non-condensing)

Housing (including Polycarbonate, splashproof and dustproof

rubber boot): Battery can be changed without removing

rubber boot.

Charging The Battery

Always fully charge the battery before using the instrument. The instrument's Li-ion battery is charged by placing the instrument in its cradle. Contacts on the bottom of the instrument meet the cradle's contacts, transferring power without other connections.

Note: Before setting the instrument into its charging cradle, visually inspect the contacts to make sure they are clean. If they are not, wipe them with a soft cloth. Do not use solvents or cleaners.

Follow this procedure to charge the instrument:

1. Plug the AC/DC adapter's barrel connector into the instrument's cradle.



- 2. Plug the AC/DC adapter into the wall outlet.
- 3. Place the instrument into the cradle, press down, and lean it back. It locks in place and the LED in the cradle glow

The instrument begins charging automatically. The "Primary" LED in the cradle blinks green to indicate charging. During charging, the diagonal lines in the battery icon on the instrument's display are animated and you see the message "Charging..."

When the instrument's battery is fully charged, the battery icon is no longer animated and shows a full battery. The message "Fully charged!" is shown. The cradle's LED glows continuously green.



Note: If you see the "Battery Charging Error" icon (a battery outline with an exclamation mark inside), check that the instrument or rechargeable battery has been set into the cradle



properly. If you still receive the message, check the Troubleshooting section of this guide.

Note: If the instrument or battery has been in the cradle for more than 10 hours and you see the "Battery Charging Error" icon and a message that says, "Charging Too Long," this indicates that the battery is not reaching a full charge. Try changing the battery and make sure the contacts between the instrument (or battery) are meeting the cradle. If the message is still shown, consult your distributor or RAE Systems Technical Services.

Charging A Spare Rechargeable Battery

A rechargeable Li-ion battery can be charged when it is not inside the monitor. The charging cradle is designed to accommodate both types of charging. Contacts on the bottom of the battery meet the contacts on the cradle, transferring power without other connections, and a spring-loaded capture holds the battery in place during charging.

- 1. Plug the AC/DC adapter into the monitor's cradle.
- 2. Place the battery into the cradle, with the gold-plated contacts on top of the six matching charging pins.
- 3. Plug the AC/DC adapter into the wall outlet.

The battery begins charging automatically. During charging, the Secondary LED in the cradle blinks green. When charging is complete, it glows steady green.

Release the battery from the cradle by pulling it back toward the rear of the cradle and tilting it out of its slot.

Note: If you need to replace the Li-ion battery pack, replacements are available from RAE Systems. The part number is 059-3051-000.

Note: An Alkaline Battery Adapter (part number 059-3052-000), which uses four AA alkaline batteries (Duracell MN1500), may be substituted for the Li-Ion battery.

WARNING!

To reduce the risk of ignition of hazardous atmospheres, recharge and replace batteries only in areas known to be non-hazardous. Remove and replace batteries only in areas known to be non-hazardous.

Low Voltage Warning

When the battery's charge falls below a preset voltage, the instrument warns you by beeping once and flashing once every minute, and the "empty battery" icon blinks on and off once per second. You should turn off the instrument within 10 minutes and either recharge the battery by placing the instrument in its cradle, or replace the battery with a fresh one with a full charge.



Clock Battery

An internal clock battery is mounted on one of the instrument's printed circuit boards. This long-life battery keeps settings in memory from being lost whenever the Li-ion battery or alkaline batteries are removed. This backup battery should last approximately five years, and must be replaced by an authorized RAE Systems service technician. It is not user-replaceable.

Data Protection While Power Is Off

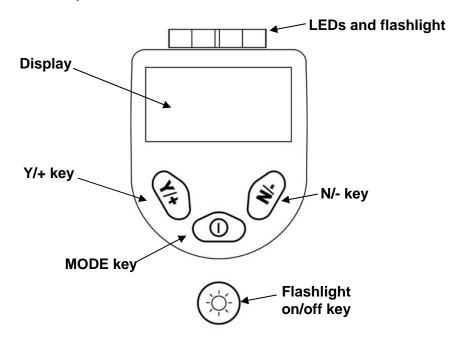
When the instrument is turned off, all the current real-time data including last measured values are erased. However, the datalog data is preserved in non-volatile memory. Even if the battery is disconnected, the datalog data will not be lost.

User Interface

The instrument's user interface consists of the display, LEDs, an alarm transducer, and four keys. The keys are:

Y/+ MODE N/-Flashlight on/off

The LCD display provides visual feedback that includes the reading, time, battery condition, and other functions.

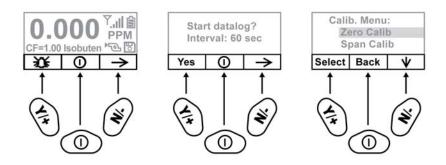


In addition to their labeled functions, the keys labeled Y/+, MODE, and N/- act as "soft keys" that control different parameters and make different selections within the instrument's menus. From menu to

menu, each key controls a different parameter or makes a different selection.

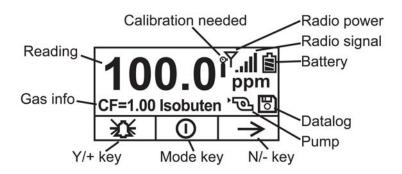
Three panes along the bottom of the display are "mapped" to the keys. These change as menus change, but at all times the left pane corresponds to the [Y/+] key, the center pane corresponds to the [MODE] key, and the right pane corresponds to the [N/-] key. Here are three examples of different menus with the relationships of the keys clearly shown:

RELATIONSHIP OF BUTTONS TO CONTROL FUNCTIONS



Display

The display shows the following information:



Graph Graphic representation of concentration plotted

over time

Gas infoTells the Correction Factor and type of

calibration gas

Reading Concentration of gas as measured by the

instrument

Calibration needed

Radio power

Indicates that calibration should be performed Indicates whether radio connection is on or

off

Radio signal Indicates signal strength in 5-bar bargraph

BatteryIndicates battery level in 3 barsPumpIndicates that pump is workingDatalogIndicates whether datalog is on or offY/+Y/+ key's function for this screen

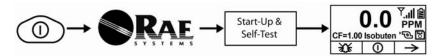
MODE key's function for this screen N/- N/- key's function for this screen

Operating The Instrument

The instrument is designed as a broadband VOC gas monitor and datalogger for work in hazardous environments. It gives real-time measurements and activates alarm signals whenever the exposure exceeds preset limits. Prior to factory shipment, the instrument is preset with default alarm limits and the sensor is pre-calibrated with standard calibration gas. However, you should test the instrument and verify the calibration before the first use. After the instrument is fully charged and calibrated, it is ready for immediate operation.

Turning The Instrument On

- 1. With the instrument turned off, press and hold [MODE].
- 2. When the display turns on, release the [MODE] key.



The RAE Systems logo should appear first. (If the logo does not appear, there is likely a problem and you should contact your distributor or RAE Systems Technical Support.) The instrument is now operating and performs self tests. If any tests (including sensor and memory tests fail), refer to the Troubleshooting section of this guide.

Once the startup procedure is complete, the instrument shows a numerical reading screen with icons. This indicates that the instrument is fully functional and ready to use.

Turning The Instrument Off

- 1. Press and hold the Mode key for 3 seconds. A 5-second countdown to shutoff begins.
- 2. Once the countdown stops, the instrument is off. Release the Mode key.
- 3. When you see "Unit off..." release your finger from the [MODE] key. The instrument is now off.

Note: You must hold your finger on the key for the entire shutoff process. If you remove your finger from the key during the countdown, the shutoff operation is canceled and the instrument continues normal operation.

Operating The Built-In Flashlight

The instrument has a built-in flashlight that helps you point the probe in dark places. Press the flashlight key to turn it on. Press it again to turn it off.

Note: Using the flashlight for extended periods shortens the battery's operating time before it needs recharging.

Pump Status

IMPORTANT!

During operation, make sure the probe inlet and the gas outlet are free of obstructions. Obstructions can cause premature wear on the pump, false readings, or pump stalling. During normal operation, the pump icon alternately shows inflow and outflow as shown here:



During duty cycling (PID lamp cleaning), the display shows these icons in alternation:



If there is a pump failure or obstruction that disrupts the pump, you will see this icon blinking on and off:



If you see this blinking icon, consult the Troubleshooting section of this guide.

Calibration Status

The instrument displays this icon if it requires calibration:



Calibration is required (and indicated by this icon) if:

- The lamp type has been changed (for example, from 10.6 eV to 9.8 eV).
- The sensor has been replaced.
- It has been 30 days or more since the instrument was last calibrated.
- If you have changed the calibration gas type without recalibrating the instrument.

Operating Modes

Your instrument operates in different modes, depending on the model and its factory default settings. In some cases, you can change modes using a password and using the instrument's navigation. In other cases, you must use ProRAE Studio software.

The default setting for your instrument is:

User Mode: Basic

Operation Mode: Hygiene

This is outlined in detail on page 74.

The other options, covered later in this guide, are:

User Mode: Advanced (page 78)
Operation Mode: Hygiene

User Mode: Advanced (page 82)

Operation Mode: Search

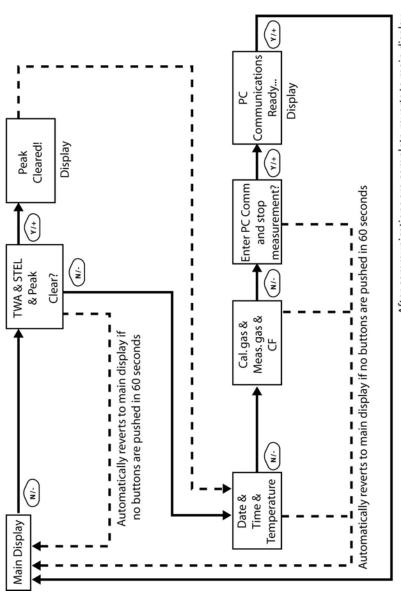
Using ProRAE Studio allows access to other options. In addition, Diagnostic Mode (page 83) is available for service technicians.

Basic User Level/Hygiene Mode (Default Settings)

The instrument is programmed to operate in Basic User Level/Hygiene Mode as its default. This gives you the most commonly needed features while requiring the fewest parameter adjustments.

Pressing [N/-] steps you from one screen to the next, and eventually return to the main display. If you do not press a key within 60 seconds after entering a display, the instrument reverts to its main display.

Note: While viewing any of these screens, you can shut off your instrument by pressing [MODE].



After communications are complete, reverts to main display

Note: Dashed line indicates automatic progression.

After the instrument is turned on, it runs through the start-up menu. Then the message "**Please apply zero gas...**" is displayed.

At this point, you can perform a zero air (fresh air) calibration. If the ambient air is clean, you can use that. Otherwise, use a cylinder of zero air. Refer to Zero Calibration on page 37 for a more detailed description of zero calibration.

Start zero calibration by pressing Start. You see the message "Zeroing..." followed by a 30-second countdown.

Note: You can press [MODE] to quit, bypassing the zero air calibration.

When zero calibration is complete, you see the message:

Zeroing is done!

Reading = 0.0 ppm

The instrument is now sampling and collecting data.

Note: At the Average & Peak, Date & Time & Temperature, Calibration Gas & Measurement Gas & Correction Factor, and PC Communications screens, the instrument automatically goes to the main display after 60 seconds if you do not push a key to make a selection.

Alarm Signals

During each measurement period, the gas concentration is compared with the programmed alarm limits (gas concentration alarm limit settings). If the concentration exceeds any of the preset limits, the loud buzzer and red flashing LED are activated immediately to warn you of the alarm condition.

In addition, the instrument alarms if one of the following conditions occurs: battery voltage falls below a preset voltage level, failure of the UV lamp, or pump stall.

Alarm Signal Summary

Message	Condition	Alarm Signal
HIGH	Gas exceeds "High Alarm" limit	3 beeps/flashes per second*
OVR	Gas exceeds measurement range	3 beeps/flashes per second*
MAX	Gas exceeds electronics' maximum range	3 beeps/flashes per second*
LOW	Gas exceeds "Low Alarm" limit	2 beeps/flashes per second*
TWA	Gas exceeds "TWA" limit	1 Beep/flash per second*
STEL	Gas exceeds "STEL" limit	1 Beep/flash per second*
Pump icon flashes	Pump failure	3 beeps/flashes per second
Lamp	PID lamp failure	3 beeps/flashes per second plus "Lamp" message on display
Battery icon flashes	Low battery	1 flash, 1 beep per minute plus battery icon flashes on display
CAL	Calibration failed, or needs calibration	1 beep/flash per second
NEG	Gas reading measures less than number stored in calibration	1 beep/flash per second

^{*} Hygiene mode only. In Search mode, the number of beeps per second (1 to 7) depends upon the concentration of the sampled gas. Faster rates indicate higher concentrations.

Preset Alarm Limits & Calibration

The instrument is factory calibrated with standard calibration gas, and is programmed with default alarm limits.

Cal Gas (Isobutylene)	Cal Span	unit	Low	High	TWA	STEL
MiniRAE 3000	100	ppm	50	100	10	25

Testing The Alarm

You can test the alarm whenever the main (Reading) display is shown. Press [Y/+], and the audible and visible alarms are tested.

Integrated Sampling Pump

The instrument includes an integrated sampling pump. This diaphragm-type pump that provides a 450 to 550 cc per minute flow rate. Connecting a Teflon or metal tubing with 1/8" inside diameter to the gas inlet port of the instrument, this pump can pull in air samples from 100' (30 m) away horizontally or vertically.

Note: In Search Mode, the pump turns on when a sample measurement is started, and turns off when the sample is manually stopped.

If liquid or other objects are pulled into the inlet port filter, the instrument detects the obstruction and immediately shuts down the pump. The alarm is activated and a flashing pump icon is displayed.

You should acknowledge the pump shutoff condition by clearing the obstruction and pressing the [Y/+] key while in the main reading display to restart the pump.

Backlight

The LCD display is equipped with an LED backlight to assist in reading the display under poor lighting conditions.

Datalogging

During datalogging, the instrument displays a disk icon to indicate that datalogging is enabled. The instrument stores the measured gas concentration at the end of every sample period (when data logging is enabled). In addition, the following information is stored: user ID, site ID, serial number, last calibration date, and alarm limits. All data are retained (even after the unit is turned off) in non-volatile memory so that it can be down-loaded at a later time to a PC.

Datalogging event

When Datalogging is enabled, measurement readings are being saved. These data are stored in "groups" or "events." A new event is created and stored each time the instrument is turned on and is set to automatic datalogging, or a configuration parameter is changed, or datalogging is interrupted. The maximum time for one event is 24 hours or 28,800 points. If an event exceeds 24 hours, a new event is automatically created. Information, such as start time, user ID, site ID, gas name, serial number, last calibration date, and alarm limits are recorded.

Datalogging sample

After an event is recorded, the unit records a shorter form of the data. When transferred to a PC running ProRAE Studio, this data is arranged with a sample number, time, date, gas concentration, and other related information.

Auto/Manual/Snapshot Datalogging

The instrument has three datalog types:

Auto Default mode. Collects datalog information when the

instrument is sampling.

Manual Datalogging occurs only when the instrument's

datalogging is manually started (see page 63 for

details).

Snapshot Datalogs only during snapshot (single-event capture,

initiated by pressing [MODE]) sampling. See page 65

for details.

Note: You can only choose one datalog type to be active at a time.

Accessories

The following accessories are included with the instrument:

- An AC Adapter (Battery Charger)
- Alkaline battery adapter
- External Filter
- Organic Vapor Zeroing kit

Hard-case kits also include these accessories:

- Calibration adapter
- Calibration regulator and Flow controller

Standard Kit & Accessories AC Adapter (Battery Charger)

WARNING

To reduce the risk of ignition of hazardous atmospheres, recharge battery only in area known to be non-hazardous. Remove and replace battery only in area known to be non-hazardous.

Ne charger les batteries que dans emplacements designés nondangereuses.

A battery charging circuit is built into the instrument cradle. It only needs a regular AC to 12 VDC adapter (wall-mount transformer, part number 500-0114-000) to charge the instrument.

To charge the battery inside the instrument:

- 1. Power off the instrument.
- 2. Connect the AC adapter to the DC jack on the instrument's cradle. If the instrument is off, it automatically turns on.
- 3. While charging, the display message shows "Charging." The Primary LED on the cradle flashes green when charging.
- 4. When the battery is fully charged, the LED changes to glowing green continuously, and the message "Fully charged" appears on the

display. If there is a charging error, the LED glows red continuously.

A completely discharged instrument can be charged to full capacity within 8 hours. Batteries drain slowly even if an instrument is off. Therefore, if the instrument has been in storage or has not been charged for several days or longer, check the charge before using it.

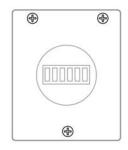
The factory-supplied battery is designed to last for 16 hours of normal operation (no alarm), for a new battery under the optimum circumstances. As the battery becomes older or is subject to adverse conditions (such as cold ambient temperature), its capacity will be significantly reduced.

Alkaline Battery Adapter

An alkaline battery adapter is supplied with each instrument. The adapter (part number 059-3052-000) accepts four AA alkaline batteries (use only Duracell MN1500) and provides approximately 12 hours of operation. The adapter is intended to be used in emergency situations when there is no time to charge the Li-ion battery pack.

To insert batteries into the adapter:

- 1. Remove the three Philips-head screws to open the compartment in the adapter.
- 2. Insert four fresh AA batteries as indicated by the polarity (+/-) markings.
- 3. Replace the cover. Replace the three screws.



To install the adapter in the instrument:

- 1. Remove the Li-ion battery pack from the instrument by sliding the tab and tilting out the battery.
- 2. Replace it with the alkaline battery adapter
- 3. Slide the tab back into place to secure the battery adapter.

IMPORTANT!

Alkaline batteries cannot be recharged. The instrument's internal circuit detects alkaline batteries and will not allow recharging. If you place the instrument in its cradle, the alkaline battery will not be recharged. The

internal charging circuit is designed to prevent damage to alkaline batteries and the charging circuit when alkaline batteries are installed inside the instrument. If you try to charge an alkaline batteries installed in the instrument, the instrument's display will say, "Alkaline Battery," indicating that it will not charge the alkaline batteries.

Note: When replacing alkaline batteries, dispose of old ones properly.

WARNING!

To reduce the risk of ignition of hazardous atmospheres, recharge the battery only in areas known to be non-hazardous. Remove and replace the battery only in areas known to be non-hazardous.

External Filter

The external filter is made of PTFE (Teflon®) membrane with a 0.45 micron pore size to prevent dust or other particles from being sucked into the sensor manifold, which would cause extensive damage to the instrument. It prolongs the operating life of the sensor. To install the external filter, simply connect it to the instrument's inlet tube.

Optional Accessories Calibration Adapter

The calibration adapter for the instrument is a simple 6-inch Tygon tubing with a metal adapter on one end. During calibration, simply insert the metal adapter into the regular gas inlet probe of the instrument and the tubing to the gas regulator on the gas bottle.

Calibration Regulator

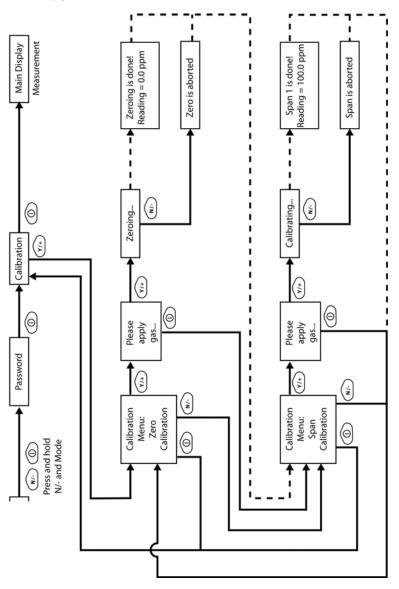
The Calibration Regulator is used in the calibration process. It regulates the gas flow rate from the Span gas cylinder into the gas inlet of the instrument during calibration process. The maximum flow rate allowed by the flow controller is about 0.5L/min (500 cc per min.). Alternatively, a demand-flow regulator or a Tedlar gas bag may be used to match the pump flow precisely.

Organic Vapor Zeroing Kit

The Organic Vapor Zeroing Kit is used for filtering organic air contaminants that may affect the zero calibration reading. To use the Organic Vapor Zeroing Kit, simply connect the filter to the inlet port of the instrument.

Standard Two-Point Calibration (Zero & Span)

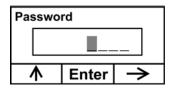
The following diagram shows the instrument's calibrations in Basic/Hygiene mode.



Note: Dashed line indicates automatic progression.

Entering Calibration

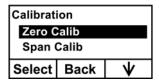
1. Press and hold [MODE] and [N/-] until you see the Password screen.



2. In Basic User Level, you do not need a password to perform calibrations. Instead of inputting a password, enter calibration by pressing [MODE].

Note: If you inadvertently press [Y/+] and change any of the numbers, simply press [MODE] and you will be directed to the calibration menu.

The Calibration screen is now visible with Zero Calibration highlighted.



These are your options:

- Press [Y/+] to select the highlighted calibration (Zero Calib or Span Calib).
- Press [MODE] to exit calibration and return to the main display and resume measurement.
- Press [N/-] to toggle the highlighted calibration type.

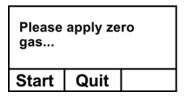
Zero (Fresh Air) Calibration

This procedure determines the zero point of the sensor calibration curve. To perform a fresh air calibration, use the calibration adapter to connect the instrument to a "fresh" air source such as from a cylinder or Tedlar bag (optional accessory). The "fresh" air is clean, dry air without organic impurities and an oxygen value of 20.9%. If such an air cylinder is not available, any clean ambient air without detectable contaminants or a charcoal filter can be used.

At the Zero Calibration menu, you can proceed to perform a Zero calibration or bypass Zero calibration and perform a Span calibration. You may also go back to the initial Calibration menu if you want to exit calibration.

- Press [Y/+] to start calibration.
- Press [MODE] to quit and return to the main calibration display.

If you have pressed [Y/+] to enter Zero calibration, then you will see this message:



- 1. Turn on your Zero calibration gas.
- 2. Press [Y/+] to start calibration.

Note: At this point, you may press [MODE] if you decide that you do not want to initiate calibration. This will take you directly to the Calibration menu, highlighted for Span calibration.

3. Zero calibration starts a 30-second countdown and displays this message:

Zeroing...

During the zeroing process, the instrument performs the Zero calibration automatically and does not require any action on your part.

Note: To abort the zeroing process at any time and proceed to Span calibration, press [N/-] at any time while zeroing is being performed. You will see a confirmation message that says "Zero aborted!" and then the Span calibration menu appears.

When Zero calibration is complete, you see this message:

Zeroing is done! Reading = 0.0 ppm

The instrument will then show the Calibration menu on its display, with Span Calib highlighted.

Span Calibration

This procedure determines the second point of the sensor calibration curve for the sensor. A cylinder of standard reference gas (span gas) fitted with a 500 cc/min. flow-limiting regulator or a flow-matching regulator is the simplest way to perform this procedure. Choose the 500 cc/min. regulator only if the flow rate matches or slightly exceeds the flow rate of the instrument pump. Alternatively, the span gas can first be filled into a Tedlar bag or delivered through a demand-flow regulator. Connect the calibration adapter to the inlet port of the instrument, and connect the tubing to the regulator or Tedlar bag.

Another alternative is to use a regulator with >500 cc/min flow but allow the excess flow to escape through a T or an open tube. In the latter method, the span gas flows out through an open tube slightly wider than the probe, and the probe is inserted into the calibration tube.

At the Span Calibration menu, you perform a Span calibration. You may also go back to the Zero calibration menu or to the initial Calibration menu if you want to exit calibration.

- Press [Y/+] to enter Span calibration.
- Press [N/-] to skip Span calibration and return to Zero calibration.
- Press [MODE] to exit Span calibration and return to the top calibration menu.

If you have pressed [Y/+] to enter Span calibration, then you will see the name of your Span gas (the default is isobutylene) and the span value in parts per million (ppm). You will also see this message that prompts you:

C. Gas = Isobutene		
Span = 100 ppm		
Please apply gas 1		
Start	Quit	

- 1. Turn on your span calibration gas.
- 2. Press [Y/+] to initiate calibration.

Note: You may press [MODE] if you decide that you do not want to initiate calibration. This will abort the span calibration and take you directly to the Calibration menu for Zero calibration.

3. Span calibration starts and displays this message:

Calibrating...

During the Span calibration process, there is a 30-second countdown and the instrument performs the Span calibration automatically. It requires no actions on your part.

Note: If you want to abort the Span calibration process, press [N/-] at any time during the process. You will see a confirmation message that says "Span is aborted!" and then the Zero calibration menu appears. You can then proceed to perform a Zero calibration, perform a Span calibration, or exit to the topmost Calibration menu.

When Span calibration is complete, you see a message similar to this (the value is an example only):

Span 1 is done! Reading = 100.0 ppm

The instrument then exits Span calibration and shows the Zero calibration menu on its display.

Note: The reading should be very close to the span gas value.

Exiting Two-Point Calibration In Basic User Level

When you are done performing calibrations, press [MODE], which corresponds with "Back" on the display. You will see the following message:

Updating settings...

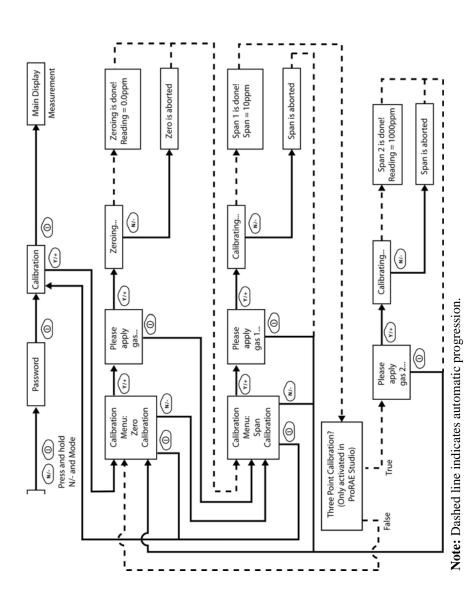
The instrument updates its settings and then returns to the main display. It begins or resumes monitoring.

Three-Point Calibration

For enhanced accuracy, it is possible to perform a second Span calibration in addition to the Zero and Span calibrations outlined in the previous section. Your instrument first must be set to allow this third calibration. This requires using ProRAE Studio software and a PC, as well as a higher concentration of calibration gas.

Note: Once the third calibration is set, you do not need to use ProRAE Studio to allow future 3-point calibrations. Also, you can only disable 3-point calibration capability by using ProRAE Studio again.

Perform the Zero and Span calibrations. After the first Span calibration (Span 1) is completed, the display a second Span calibration (Span 2) can be performed. The process is identical to the first calibration. As in the Span 1 calibration, you may exit and return to the Zero calibration screen if you choose not to perform this calibration or to abort it.



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Span 2 Calibration

A cylinder of standard reference gas (span gas) fitted with a 500 cc/min. flow-limiting regulator or a flow-matching regulator is the simplest way to perform this procedure.

Note: This gas should be of a higher concentration than the gas used for Span 1 calibration.

Choose the 500 cc/min. regulator only if the flow rate matches or slightly exceeds the flow rate of the instrument pump. Alternatively, the span gas can first be filled into a Tedlar bag or delivered through a demand-flow regulator. Connect the calibration adapter to the inlet port of the instrument, and connect the tubing to the regulator or Tedlar bag.

Another alternative is to use a regulator with >500 cc/min flow but allow the excess flow to escape through a T or an open tube. In the latter method, the span gas flows out through an open tube slightly wider than the probe, and the probe is inserted into the calibration tube.

At the Span Calibration menu, you perform a Span calibration. You may also go back to the Zero calibration menu or to the initial Calibration menu if you want to exit calibration.

- Press [Y/+] to enter Span 2 calibration.
- Press [N/-] to skip Span calibration and return to Zero calibration.
- Press [MODE] to exit Span calibration and return to the top calibration menu.

If you have pressed [Y/+] to enter Span calibration, then you will see the name of your Span gas (the default is isobutylene) and the span value in parts per million (ppm). You will also see this message that prompts you:

Please apply gas...

- 4. Turn on your span calibration gas.
- 5. Press [Y/+] to initiate calibration.

Note: You may press [MODE] if you decide that you do not want to initiate calibration. This will take you directly to the Calibration menu for Zero calibration.

6. Span calibration starts a 30-second countdown and displays this message:

Calibrating...

During the Span calibration process, the instrument performs the Span calibration automatically and does not require any action on your part.

Note: If you want to abort the Span calibration process, press [N/-] at any time during the process. You will see a confirmation message that says "Span is aborted!" and then the Zero calibration menu will appear. You can then proceed to perform a Zero calibration, perform a Span calibration, or exit to the topmost Calibration menu.

When Span calibration is complete, you will see a message similar to this (the value shown here is for example only):

Span 2 is done! Reading = 1000 ppm

The instrument then exits Span calibration and shows the Zero calibration menu on its display.

Note: The reading should be very close to the span gas value.

Exiting Three-Point Calibration

When you are done performing calibrations, press [MODE], which corresponds with "Back" on the display. You will see the following message:

Updating settings...

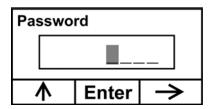
The instrument updates its settings and then returns to the main display. It begins or resumes monitoring.

Programming Mode

Programming Mode can be entered from either Hygiene Mode or Search Mode. If the current user mode is Basic, you must provide a 4-digit password to enter.

Entering Programming Mode

1. Press and hold [MODE] and [N/-] until you see the Password screen.



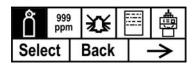
- 2. Input the 4-digit password:
 - Increase the number from 0 through 9 by pressing [Y/+].
 - Step from digit to digit using [N/-].
 - Press [MODE] when you are done.

If you make a mistake, you can cycle through the digits by pressing [N/-] and then using [Y/+] to change the number in each position.

Note: The default password is 0000.

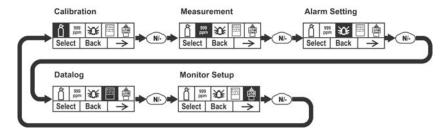
When you have successfully entered Programming Mode, you see this screen:

Calibration



Note: The password can only be changed by connecting the instrument to a PC running ProRAE Studio software. Follow the instructions in ProRAE Studio to change it.

The Calibration label is shown and its icon is highlighted, but you can press [N/-] to step from one programming menu to the next, with the name of the menu shown at the top of the display and the corresponding icon highlighted. As you repeatedly press [N/-], the selection moves from left to right, and you see these screens:



Note: When you reach Monitor Setup and press [N/-], the menu cycles back to Calibration.

Programming Mode Menus

The Programming Mode allows anyone with the password to change the instrument's settings, calibrate the instrument, modify the sensor configuration, enter user information, etc. Programming Mode has five menus. Each menu includes several sub-menus to perform additional programming functions.

This table shows the menus and sub-menus:

Å	999 ppm	狱		
Calibration	Measurement	Alarm Setting	Datalog	Monitor Setup
Zero Calibration	Meas. Gas	High Alarm	Clear Datalog	Op Mode
Span Calibration	Meas. Unit	Low Alarm	Interval	Site ID
		STEL Alarm	Data Selection	User ID
		TWA Alarm	Datalog Type	User Mode
		Alarm Type		Date
		Buzzer & Light		Time
				Pump Duty Cycle
				Pump Speed
				Temperature Unit
				Language Radio
				Power
				Real Time Protocol
				Power On
				Zero
				Unit ID LCD
				Contrast

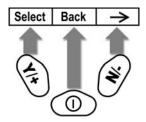
Once you enter Programming Mode, the LCD displays the first menu, Calibration. Each subsequent menu is accessed by pressing [N/-] repeatedly until the desired menu is displayed. To enter a sub-menu of a menu, press [Y/+].

Exiting Programming Mode

To exit Programming Mode and return to normal operation, press [MODE] once at any of the programming menu displays. You will see "Updating Settings..." as changes are registered and the mode changes.

Navigating Programming Mode Menus

Navigating through the Programming Mode menus is easy and consistent, using a single interface format of "Select," "Back" and "Next" at the top level. The three control buttons correspond to these choices as shown:



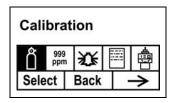
Note: Pressing [MODE] in the Programming Mode's top level causes the instrument to exit Programming Mode and return to monitoring.

The three keys perform the following functions in Programming Mode:

Key	Function in Programming Mode
[MODE]:	Exit menu when pressed momentarily or exit data entry mode
[Y/+]:	Increase alphanumerical value for data entry or confirm (yes) for a question
[N/-]:	Provides a "no" response to a question

Calibration

Two types of calibration are available: Zero (fresh air) and Span.



Select Zero or Span Calibration by pressing [N/+]. Once your choice is highlighted, press [Y/+].

Zero Calibration

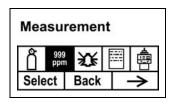
The procedure for performing a zero calibration is covered on page 35.

Span Calibration

The procedure for performing a basic span calibration is covered on page 35.

Measurement

The sub-menus for Measurement are Measurement Gas and Measurement Unit.



Meas. Gas

Measurement gases are organized in four lists:

- My List is a customized list of gases that you create. It contains a maximum of 10 gases and can only be built in ProRAE Studio on a PC and transferred to the instrument. **Note:** The first gas in the list is always isobutylene (it cannot be removed from the list).
- Last Ten is a list of the last ten gases used by your instrument. The list is built automatically and is only updated if the gas selected from Custom Gases or Library is not already in the Last Ten. This ensures that there is no repetition.
- Gas Library is a library that consists of all the gases found in RAE Systems' Technical Note TN-106 (available online at www.raesystems.com).
- Custom Gases are gases with user-modified parameters. Using ProRAE Studio, all parameters defining a gas can be modified, including the name, span value(s), correction factor, and default alarm limits.
 - 1. Scroll through each list by pressing [N/-].
 - 2. Press [Y/+] to select one (My List, Last Ten, Gas Library, or Custom Gases).

- 3. Once you are in one of the categories, press [N/-] to scroll through its list of options and [Y/+] to select one. (If you press [MODE], you exit to the next submenu.)
- 4. Press [Y/+] to save your choice or [N/-] to undo your selection.

Leave the sub-menu and return to the Programming Mode menus by pressing [MODE].

Meas. Unit

Standard available measurement units include:

Abbreviation	Unit	MiniRAE 3000
ppm	parts per million	Yes
ppb	parts per billion	
mg/m3	milligrams per cubic meter	Yes
ug/m3	micrograms per cubic meter	

- Scroll through the list by pressing [N/-].
- Select by pressing [Y/+].
- Save your selection by pressing [Y/+] or undo your selection by pressing [N/-].

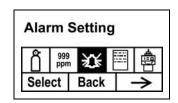
Leave the sub-menu and return to the Programming Mode menus by pressing [MODE].

Alarm Setting

During each measurement period, the gas concentration is compared with the programmed alarm limits (gas concentration alarm limit settings: Low, High, TWA and STEL). If the concentration exceeds any of the preset limits, the loud buzzer and red flashing LED are activated immediately to warn of the alarm condition.

An alarm signal summary is shown on page 27.

In this menu, you can change the High and Low alarm limits, the STEL limit, and the TWA. Press [Y/+] to to enter the Alarm Setting menu. **Note:** All settings are shown in ppb (parts per billion), or $\mu g/m^3$ (micrograms per cubic meter), depending on your setting.



- 1. Scroll through the Alarm Limit sub-menu using the [N/-] key until the display shows the desired limit to be changed (High Alarm, Low Alarm, STEL Alarm, and TWA Alarm)
- 2. Press [Y/+] to select one of the alarm types. The display shows a flashing cursor on the left-most digit of the previously stored alarm limit.
- 3. Press [Y/+] to increase each digit's value.
- 4. Press [N/-] to advance to the next digit.
- 5. Again, use [Y/+] to increase the number.

Repeat this process until all numbers are entered.

Press [MODE] when you are done.

- Press [Y/+] to save the changes.
- Press [N/-] to undo the changes and revert to the previous settings. When all alarm types have been changed or bypassed, press [MODE] to exit to the Programming Menu.

High Alarm

You can change the High Alarm limit value. The value is typically set by the instrument to match the value for the current calibration gas. It is expressed in parts per billion (ppb). **Note:** The default value depends on the measurement gas.

To change the High Alarm value:

- 1. Press [Y/+] to increase each digit's value.
- 2. Press [N/-] to advance to the next digit.
- 3. Again, use [Y/+] to increase the number.

Repeat this process until all numbers are entered.

When you have completed your selections, press [MODE]. You will see two choices: Save and Undo. You have the opportunity to register the new settings or to change your mind and revert to your previous settings.

Press [Y/+] to save the changes.

Press [N/-] to undo the changes and revert to the previous settings.

Low Alarm

You can change the Low Alarm limit value. The value is typically set by the instrument to match the value for the current calibration gas. It is expressed in parts per billion (ppb). **Note:** The default value depends on the measurement gas.

To change the Low Alarm value:

- 1. Press [Y/+] to increase each digit's value.
- 2. Press [N/-] to advance to the next digit.
- 3. Again, use [Y/+] to increase the number.

Repeat this process until all numbers are entered.

When you have completed your selections, press [MODE]. You will see two choices: Save and Undo. You have the opportunity to register the new settings or to change your mind and revert to your previous settings.

- Press [Y/+] to save the changes.
- Press [N/-] to undo the changes and revert to the previous settings.

STEL Alarm

You can change the STEL Alarm limit value. The value is typically set by the instrument to match the value for the calibration gas. It is expressed in parts per billion (ppb). **Note:** The default value depends on the measurement gas.

To change the STEL Alarm value:

- 1. Press [Y/+] to increase each digit's value.
- 2. Press [N/-] to advance to the next digit.
- 3. Again, use [Y/+] to increase the number.

Repeat this process until all numbers are entered.

When you have completed your selections, press [MODE]. You will see two choices: Save and Undo. You have the opportunity to register the new settings or to change your mind and revert to your previous settings.

- Press [Y/+] to save the changes.
- Press [N/-] to undo the changes and revert to the previous settings.

TWA Alarm

You can change the TWA (time-weighted average) Alarm limit value. The value is typically set by the instrument to match the value for the calibration gas. It is expressed in parts per billion (ppb). **Note:** The default value depends on the measurement gas.

To change the TWA Alarm value:

- 1. Press [Y/+] to increase each digit's value.
- 2. Press [N/-] to advance to the next digit.
- 3. Again, use [Y/+] to increase the number.

Repeat this process until all numbers are entered.

When you have completed your selections, press [MODE]. You will see two choices:

- Save
- Undo

You have the opportunity to register the new settings or to change your mind and revert to your previous settings.

- Press [Y/+] to save the changes.
- Press [N/-] to undo the changes and revert to the previous settings.

Alarm Type

There are two selectable alarm types:

Latched When the alarm is triggered, you can

manually stop the alarm.

The latched setting only controls alarms for High Alarm, Low Alarm, STEL Alarm,

and TWA alarm.

Note: To clear an alarm when the

instrument is set to "Latched," press [Y/+] when the main (Reading) display is shown.

Automatic Reset When the alarm condition is no longer

present, the alarm stops and resets itself.

1. Press [N/-] to step from one alarm type to the other.

2. Press [Y/+] to select an alarm type.

When you have completed your selections, press [MODE].

You will see two choices: Save and Undo. You have the opportunity to register the new settings or to change your mind and revert to your previous settings.

- Press [Y/+] to save the changes.
- Press [N/-] to undo the changes and revert to the previous settings.

Buzzer & Light

The buzzer and light alarms can be programmed to be on or off individually or in combination. Your choices are:

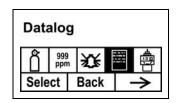
- Both on
- Light only
- Buzzer only
- Both off
- 1. Press [N/-] to step from one option to the next.
- 2. Press [Y/+] to make your selection (the dark circle in the "radio button" indicates your selection).
- 3. When you have completed your selections, press [MODE].

You will see two choices: Save and Undo. You have the opportunity to register the new settings or to change your mind and revert to your previous settings.

- Press [Y/+] to save the changes.
- Press [N/-] to undo the changes and revert to the previous settings.

Datalog

The instrument calculates and stores the concentration and ID of each sample taken. In the datalog sub-menu, a user can perform the tasks and functions shown below.



1. Scroll through the Datalog sub-menu using the [N/-] key until the display shows the desired parameter to be changed:

Clear Datalog Interval Data Selection Datalog Type

2. Press [Y/+] to make your selection. Exit by pressing [MODE] for Back

Clear Datalog

This erases all the data stored in the datalog.

Note: Once the datalog is cleared, the data cannot be recovered.

Press [Y/+] to clear the datalog. The display asks, "Are you sure?"

- Press [Y/+] if you want to clear the datalog. When it has been cleared, the display shows "Datalog Cleared!"
- Press [N/-] if you do not want to clear the datalog.

The display changes, and you are taken to the next sub-menu, Interval.

Interval

Intervals are shown in seconds. The default value is 60 seconds. The maximum interval is 3600 seconds.

- 1. Press [Y/+] to increase each digit's value.
- 2. Press [N/-] to advance to the next digit.
- 3. Again, use [Y/+] to increase the number.

Repeat this process until all numbers are entered.

When you have completed your selections, press [MODE].

You will see two choices: Save and Undo. You have the opportunity to register the new settings or to change your mind and revert to your previous settings.

- Press [Y/+] to save the changes.
- Press [N/-] to undo the changes and revert to the previous settings.

Data Selection

Data Selection allows you to select which types of data are stored and made available when you offload your datalog to a computer via ProRAE Studio software.

You can choose any or all of three types of data (you must choose at least one):

- Average
- Maximum
- Minimum
- 1. Press [N/-] to step from one option to the next. The highlighter indicates your choice.
- 2. Press [Y/+] to toggle your selection on or off (the check box indicates "on" with an "X").
- 3. When you have completed your selections, press [MODE].

You will see two choices: Save and Undo. You have the opportunity to register the new settings or to change your mind and revert to your previous settings.

- Press [Y/+] to save the changes.
- Press [N/-] to undo the changes and revert to the previous settings.

Datalog Type

The instrument has three datalog types:

Auto Default mode. Collects datalog information when the

instrument is sampling.

Manual Datalogging occurs only when the instrument's

datalogging is manually started (see below for details).

Snapshot Datalogs only during single-event capture sampling. **Note:** You can only choose one datalog type to be active at a time.

1. Press [N/-] to step from one option to the next.

- 2. Press [Y/+] to make your selection (the dark circle in the "radio button" indicates "on").
- 3. When you have completed your selection, press [MODE].

You will see two choices: Save and Undo. You have the opportunity to register the new settings or to change your mind and revert to your previous settings.

• Press [Y/+] to save the changes.

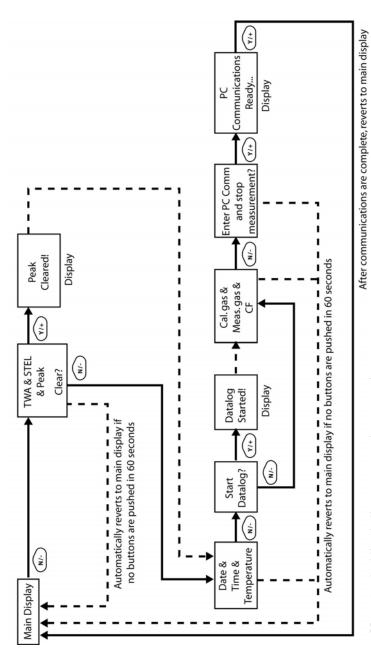
Press [N/-] to undo the changes and revert to the previous settings.

Manual Datalog

When the instrument is set to Manual Datalog, you turn datalogging on and off by stepping through the displays from the Main Display, and then pressing the keys to select datalog on/off functions.

• When you reach the screen that says "Start Datalog?" press [Y/+] to start it. You see "Datalog Started," confirming that datalogging is now on.

When you reach the screen that says "Stop Datalog?" press [Y/+] to stop it. You see "Datalog Stopped," confirming that datalogging is now off.



Note: Dashed line indicates automatic progression.

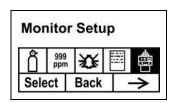
Snapshot Datalog

When the instrument is in Snapshot datalogging mode, it captures a single "snapshot" of the data at the moment of your choosing. Whenever the instrument is on and it is set to Snapshot, all you have to do is press [MODE] each time you want to capture a snapshot of the data at that instant.

When you send the data to a computer using ProRAE Studio, the data snapshots are uniquely identified by time and other parameters.

Monitor Setup

Many settings can be accessed in this menu, including setting the date and time and adjusting the pump's on/off duty cycle.



Op Mode

Under Monitor Setup is "Op Mode."

Press [Y/+] to select.

You see two options (one is highlighted):

Hygiene Search

The current mode is indicated by a dark circle within the circle in front of either Hygiene or Search.

- 1. Select Hygiene or Search by pressing [N/-]. The highlighting changes from one to the other each time you press [N/-].
- 2. Press [Y/+] to select that mode for the instrument.
- 3. Press [MODE] when you want to register your selection to place the instrument in the selected mode.
- 4. Press [Y/+] to commit the change and exit to the Monitor Setup screen, or press [N/-] to Undo (exit to the Monitor Setup screen without changing the Mode).

Site ID

Enter an 8-digit alphanumeric/character Site ID in the programming mode. This Site ID is included in the datalog report.

- 1. Press [Y/+] and the display shows the current site ID. Example: "RAE00001." Note that the left-most digit flashes to indicate it is the selected one.
- 2. Press [Y/+] to step through all 26 letters (A to Z) and 10 numerals (0 to 9).

Note: The last four digits must be numerals.

3. Press [N/-] to advance to the next digit. The next digit to the right flashes.

Repeat this process until all eight digits of the new site ID are entered.

Press [MODE] to exit.

If there is any change to the existing site ID, the display shows "Save?" Press [Y/+] to accept the new site ID. Press [N/-] to discard the change and move to the next sub-menu.

User ID

Enter an 8-digit alphanumeric User ID in the programming mode. This User ID is included in the datalog report.

- Press [Y/+] and the display shows the current User ID.
 Example: "RAE00001." Note that the left-most digit flashes to indicate it is the selected one.
- 2. Press [Y/+] to step through all 26 letters (A to Z) and 10 numerals (0 to 9).
- 3. Press [N/-] to advance to the next digit. The next digit to the right flashes.

Repeat this process until all eight digits of the new User ID are entered.

Press [MODE] to exit.

If there is any change to the existing User ID, the display shows "Save" Press [Y/+] to accept the new site ID. Press [N/-] to discard (undo) the change and move to the next sub-menu.

User Mode

The instrument has two user modes:

Basic Basic users can only see and use a basic set of functions.

Advanced Advanced users can see all screens and perform all available functions.

Note: The default value for User Mode is Basic.

To change the User Mode:

- 1. Press [N/-] to step from one option to the next. The highlighting changes each time you press [N/-].
- 2. Press [Y/+] to make your selection (the dark circle in the "radio button" indicates "on").
- 3. When you have completed your selection, press [MODE].
- 4. Press [Y/+] to accept the new User Mode. Press [N/-] to discard the change and move to the next sub-menu.

Date

The Date is expressed as Month/Day/Year, with two digits for each.

- 1. Press [Y/+] and the display shows the current date. Note that the left-most digit flashes to indicate it is selected.
- 2. Press [Y/+] to step through all 10 numerals (0 to 9).
- 3. Press [N/-] to advance to the next digit. The next digit to the right flashes.

Repeat this process until all six digits of the new date are entered.

Press [MODE] to exit.

- Press [Y/+] to save the new date.
- Press [N/-] to undo the change and move to the next sub-menu.

Time

The Time is expressed as Hours/Minutes/Seconds, with two digits for each. The time is in 24-hour (military) format.

- 1. Press [Y/+] and the display shows the current time. Note that the left-most digit flashes to indicate it is selected.
- 2. Press [Y/+] to step through all 10 numerals (0 to 9).

3. Press [N/-] to advance to the next digit. The next digit to the right flashes.

Repeat this process until all six digits of the new time are entered.

Press [MODE] to exit.

- Press [Y/+] to save the new date.
- Press [N/-] to undo the change and move to the next sub-menu.

Duty Cycle

The pump's duty cycle is the ratio of its on time to off time. The duty cycle ranges from 50% to 100% (always on), and the period is 10 seconds. Therefore, a duty cycle of 60% means that the pump is on for 6 seconds and off for four seconds. Duty cycling is employed by the instrument to clean the PID. A lower duty cycle has a greater effect on keeping the PID clean than a higher duty cycle.

Important! Pump duty cycling is interrupted when the instrument senses a gas. The pump's duty cycle is disabled when the measurement is greater than the 2ppm threshold and is re-enabled when the reading falls below 90% of the threshold (1.8 ppm).

- 1. Press [Y/+] to increase the value.
- 2. When you have completed your selection, press [MODE].
 - Press [Y/+] to save the new duty cycle value.
 - Press [N/-] to undo the change and move to the next sub-menu.

Temperature Unit

The temperature display can be switched between Fahrenheit and Celsius units.

- 1. Press [N/-] to step from one option to the next.
- 2. Press [Y/+] to make your selection (the dark circle in the "radio button" indicates "on").
- 3. When you have completed your selection, press [MODE].
 - Press [Y/+] to save the new temperature unit.
 - Press [N/-] to undo the change and move to the next sub-menu.

Pump Speed

The pump can operate at two speeds, high and low. Running at low speed is quieter and conserves a small amount of power. There is almost no difference in sampling accuracy.

- 1. Press [N/-] to step from one option to the next.
- 2. Press [Y/+] to make your selection (the dark circle in the "radio button" indicates "on").
- 3. When you have completed your selection, press [MODE].
 - Press [Y/+] to save the new temperature unit.
 - Press [N/-] to undo the change and move to the next sub-menu.

Language

English is the default language, but other languages can be selected for the instrument.

- 1. Press [N/-] to step from one option to the next.
- 2. Press [Y/+] to make your selection (the dark circle in the "radio button" indicates "on").
- 3. When you have completed your selection, press [MODE].
 - Press [Y/+] to save your new language choice.
 - Press [N/-] to undo it and return to the previous language selection.

Radio Power

The radio connection can be turned on or off.

- 1. Press [N/-] to step from one option to the next (on or off).
- 2. Press [Y/+] to make your selection (the dark circle in the "radio button" indicates that the option is selected).
- 3. When you have completed your selection, press [MODE].
 - Press [Y/+] to accept the new radio setting (on or off).
 - Press [N/-] to discard the change and move to the next submenu.

Real Time Protocol

Real Time Protocol is the setting for data transmission.

The choices are:

P2M (cable) Point to multipoint. Data is transferred from the

instrument to multiple locations using a wired

connection. Default data rate: 19200 bps.

P2P (cable) Point to point. Data is transferred only between the

instrument and one other location, such as a

computer. Default data rate: 9600 bps.

P2M (wireless) Point to multipoint, wireless. Data is transferred

wirelessly and can be received by multiple

receivers.

1. Press [N/-] to step from one option to the next.

- 2. Press [Y/+] to make your selection (the dark circle in the "radio button" indicates "on").
- 3. When you have completed your selection, press [MODE].
 - Press [Y/+] to save the new real-time communications protocol.
 - Press [N/-] to undo the change and move to the next sub-menu.

Power On Zero

When Power On Zero is on, the instrument performs a zero calibration when it is turned on.

- 1. Press [N/-] to step from one option to the next.
- 2. Press [Y/+] to make your selection (the dark circle in the "radio button" indicates your selection).
- 3. When you have completed your selection, press [MODE].
 - Press [Y/+] to save the change.
 - Press [N/-] to discard the change and move to the next submenu.

Unit ID

This three-digit number keeps data separated by instrument when more than one instrument is used in a network. If multiple sensing units are attempting to communicate with the same Host, then the units must all have a different Unit ID.

- 1. Press [Y/+] to step through all 10 numerals (0 to 9). If you pass the numeral you want, keep pressing [Y/+]. After it counts up to 9, it starts counting up from 0 again.
- 2. Press [N/-] to advance to the next digit. The next digit to the right flashes.

Repeat this process until all three digits of the Unit ID are entered.

- 3. Press [MODE] when you are done.
 - Press [Y/+] to save the change.
 - Press [N/-] to discard the change and move to the next submenu.

LCD Contrast

The display's contrast can be increased or decreased from its default setting. You may not need to ever change the default setting, but sometimes you can optimize the display to suit extreme temperature and ambient brightness/darkness conditions.

- The minimum value is 20.
- The maximum value is 60.
- 1. Press [Y/+] to increase the value or [N/-] to decrease the value.
- 2. Press [MODE] to save your selection.
 - Press [Y/+] to save your new contrast value.
 - Press [N/-] to undo it and return to the previous value.

Hygiene Mode

The instrument usually operates in Hygiene Mode, which provides basic functionality. However, it is possible to operate it in a second mode called Search Mode. Here are the primary differences:

Hygiene Mode: Automatic measurements, continuously running

and datalogging, and calculates additional

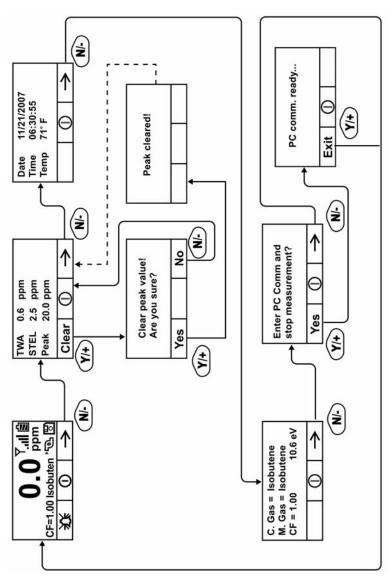
exposure values.

Search Mode: Manual start/stop of measurements and display

of certain exposure values.

Basic User Level & Hygiene Mode

The default setting is navigated in the following way:



Note: Dashed line indicates automatic progression.

Pressing [N/-] steps you from screen to screen. Options include clearing the Peak value and turning on the instrument's PC Communications for data transfer to a PC.

Entering Search Mode From Hygiene Mode

In order to change the instrument's operational mode from Hygiene Mode to Search Mode, you must enter the password-protected Programming Mode:

- 1. Hold [MODE] and [N/-] until you see the password screen.
- 2. Use [Y/+] to increment to the number you want for the first digit. (If you pass by the desired number, press [Y/+] until it cycles through to 0 again. Then press [Y/+] until you reach the desired number.)
- 3. Press [N/-] to advance to the next digit.
- 4. Again press [Y/+] to increment the number.
- 5. Press [N/-] to advance to the next digit.

Continue the process until all four numbers of the password have been input. Then press [MODE] to proceed.

The screen changes to icons with the label "Calibration."

- 1. Press [N/-] to advance to "Monitor Setup."
- 2. Press [Y/+] to select Monitor Setup.

Under Monitor Setup, you will see "Op Mode."

Press [Y/+] to select.

You will see:

Hygiene Search

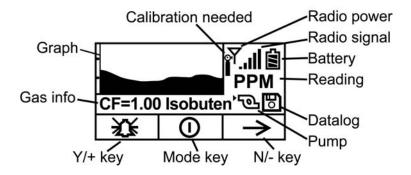
The current mode is indicated by a dark circle within the circle in front of either Hygiene or Search.

- 1. Select Hygiene or Search by pressing [N/-].
- 2. Press [Y/+] to place the instrument into the selected mode.

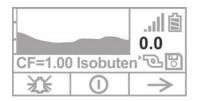
- 3. Press [MODE] when you want to register your selection to place the instrument in the selected mode.
- 4. Press [Y/+] to commit the change and exit to the Monitor Setup screen, or press [N/-] to Undo (exit to the Monitor Setup screen without changing the Mode).

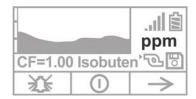
Optional Graphic Screen In Search Mode

Using ProRAE Studio, you can set your instrument to show a graphic display instead of a numeric display of ongoing data. Consult your ProRAE Studio disc for information.



During sampling, the display's readings are shown numerically, plus the graph tracks the highest readings over time. The numeric reading alternates between the value and the measurement units, as well:





Advanced User Level (Hygiene Mode Or Search Mode)

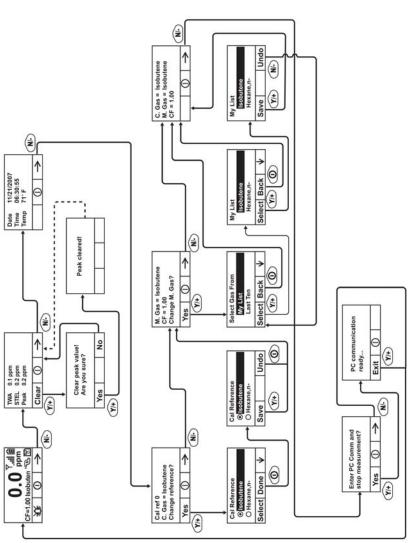
The User Mode called Advanced User Level allows a greater number of parameters to be changed than Basic User Level. It can be used with either of the Operation Modes, Hygiene Mode or Search Mode.

Advanced User Level & Hygiene Mode

With the instrument in Operation Mode: Hygiene Mode, enter User Mode: Advanced User Level (refer to the section called Monitor Mode for instructions).

Once you are in Advanced User Level and Hygiene Mode together, you can change the calibration reference and measurement gas, in addition to performing normal monitoring functions.

Pressing [N/-] progresses through the screens, while pressing [Y/+] selects options. Pressing [MODE] makes menu choices when it is shown for "Done" or "Back." Pressing and holding [Mode] whenever the circle with a vertical line in the middle is shown activates the countdown to shutoff.

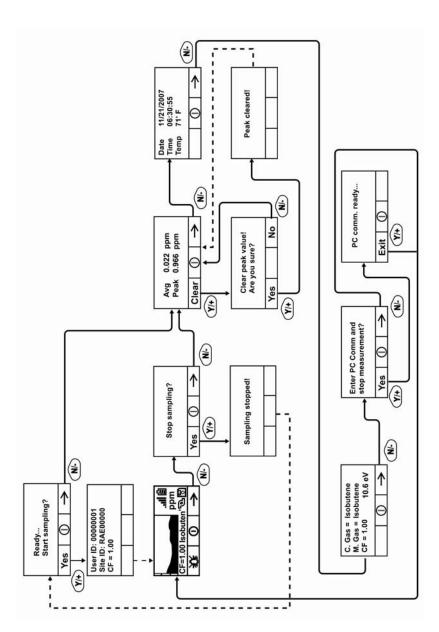


Note: Dashed line indicates automatic progression.

Basic User Level & Search Mode

With the instrument in Operation Mode: Search Mode, enter User Mode and select Basic User Level (refer to the section called User Mode for instructions).

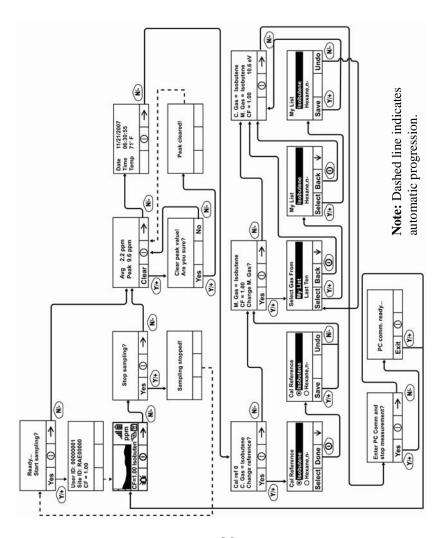
When the instrument is in Search Mode, it only samples when you activate sampling. When you see the display that says, "Ready...Start sampling?" press [Y/+] to start. The pump turns on and the instrument begins collecting data. To stop sampling, press [N/-] while the main display is showing. You will see a new screen that says, "Stop sampling?" Press [Y/+] to stop sampling. Press [N/-] if you want sampling to continue.



Note: Dashed line indicates automatic progression.

Advanced User Level & Search Mode

With the instrument in Operation Mode: Search Mode, enter User Mode and select Advanced User Level (refer to the section called Monitor Mode for instructions). Operation is similar to Basic User Level & Sampling Mode, but now allows you to change calibration and measurement reference gases. Refer to the section on measurement gases on page 52 for more details.



Diagnostic Mode

IMPORTANT! Diagnostic Mode is designed for servicing and manufacturing, and therefore is not intended for everyday use, even by advanced users. It provides raw data from sensors and about settings, but only allows adjustment of pump stall parameters, which should only be changed by qualified personnel.

Note: If the instrument is turned on in Diagnostic Mode and you switch to User Mode, datalog data remains in raw count form. To change to standard readings, you must restart the instrument.

Entering Diagnostic Mode

Note: To enter Diagnostic Mode, you must begin with the instrument turned off.

Press and hold [Y/+] and [MODE] until the instrument starts.

The instrument goes through a brief startup, and then displays raw data for the PID sensor. These numbers are raw sensor readings without calibration. The instrument is now in Diagnostic Mode.

Note: In Diagnostic Mode, the pump and lamp are normally on.

You can enter Programming Mode and calibrate the instrument as usual by pressing both [MODE] and [N/-] for three seconds.

You can enter Monitoring Mode by pressing [MODE] and [Y/+] together for three seconds.

Once the instrument is started up in Diagnostic Mode, you can switch between Diagnostic Mode and Monitoring Mode by pressing and holding [MODE] and [Y/+] simultaneously for two seconds.

In Diagnostic mode, you can step through parameter screens by pressing [MODE].

Adjusting The Pump Stall Threshold

If the gas inlet is blocked but the pump does not shut down, or the pump shuts down too easily with a slight blockage, the pump stall threshold value may be set too high or too low.

Use the following steps to adjust the pump stall threshold:

Pump High

In Diagnostic Mode, press the [MODE] key until "Pump High" is displayed. The display shows the maximum, minimum, and stall values for the pump at its high speed. Write down the "Max" reading.

Block the gas inlet and watch the pump current reading (labeled "I") increase. Write down its blocked reading. **Note:** If the pump current reading does not increase significantly (less than 10 counts), then there may be a leak in the gas inlet or the pump is weak or defective.

Add the two readings you wrote down. This is the average of the maximum block count and the maximum idle count. Divide that number by 2. Use the [Y/+] or [N/-] key to increase or decrease the stall value to equal that number.

Press the [MODE] key to exit this display.

Pump Low

In Diagnostic Mode, press the [MODE] key until "Pump Low" is displayed. The display shows the maximum, minimum, and stall values for the pump at its low speed. Write down the "Max" reading.

Block the gas inlet and watch the pump current reading (labeled "I") increase. Write down its blocked reading. **Note:** If the pump current reading does not increase significantly (less than 10 counts), then there may be a leak in the gas inlet or the pump is weak or defective.

Add the two readings you wrote down. This is the average of the maximum block count and the maximum idle count. Divide that

number by 2. Use the [Y/+] or [N/-] key to increase or decrease the stall value to equal that number.

Press the [MODE] key to exit this display.

Exiting Diagnostic Mode

You can exit Diagnostic Mode and go directly to Programming Mode or Monitor Mode as outlined above, or you can exit Diagnostic Mode completely.

To exit Diagnostic Mode so that it cannot be re-entered without a restart:

Shut down the instrument. When it is off, restart it by holding the [MODE] key. Diagnostic Mode cannot be entered until the instrument is restarted as outlined in "Entering Diagnostic Mode."

Transferring Data To & From A Computer

Once you have connected your instrument cradle to the PC, you can can transfer data, including a download of the datalog to the computer and updates of firmware to the instrument (should this ever be necessary).

Downloading The Datalog To A PC

- 1. Connect the data cable to the PC and the cradle.
- 2. Place the instrument into its cradle. The charging LED should be illuminated.
- 3. Start ProRAE Studio on your PC.
- 4. From ProRAE Studio, select "Operation" and select Setup Connection.
- 5. Select the COM port to establish a communication link between the PC and the instrument.
- 6. To receive the datalog in the PC, select "Downlog Datalog."
- 7. When you see "Unit Information," click OK.

During the data transfer, the display shows a progress bar.

When the transfer is done, you will see a screen with the datalog information. You can now export this datalog for other use or printing.

Uploading Firmware To The instrument From A PC

Uploading new firmware to your instrument requires connecting the instrument and PC. Follow these steps to make the connection:

- 1. Connect the data cable to the PC and the cradle.
- 2. Place the instrument into its cradle. The charging LED should be illuminated.
- 3. Start RAEProgrammer 7000 on your PC.
- 4. From RAEProgrammer 7000, select "Operation" and select Setup Connection.
- 5. Select the COM port to establish a communication link between the PC and the instrument.
- 6. Select Operation → Download Firmware.

Once communication is established, follow the instructions that accompany RAEProgrammer 7000 and the firmware to upload the new firmware to your instrument.

Note: Check for the latest updates to ProRAEProgrammer 7000 at www.raesystems.com.

Maintenance

The major maintenance items of the instrument are:

- Battery pack
- Sensor module
- PID lamp
- Sampling pump
- Inlet connectors and filters

Note: Maintenance should be performed by qualified personnel only.

NOTE: The printed circuit board of the instrument is connected to the battery pack even if the power is turned off. Therefore, it is very important to disconnect the battery pack before servicing or replacing any components inside the instrument. Severe damage to the printed circuit board or battery may occur if the battery pack is not disconnected before servicing the unit.

Battery Charging & Replacement

When the display shows a flashing empty battery icon, the battery requires recharging. It is recommended to recharge the instrument upon returning from fieldwork. A fully charged battery runs a instrument for 16 hours continuously. The charging time is less than 8 hours for a fully discharged battery. The battery may be replaced in the field (in areas known to be non-hazardous), if required.

WARNING!

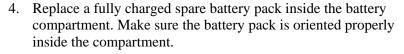
To reduce the risk of ignition of hazardous atmospheres, recharge battery only in area known to be non-hazardous. Remove and replace battery only in areas known to be non-hazardous.

Replacing The Li-ion Battery

- 1. Turn off the instrument.
- 2. Located on the rear of the instrument is a battery tab. Slide it down to unlock the battery.



3. Remove the battery pack from the battery compartment by tilting it out.



5. Slide the capture tab back up to its locked position.

Replacing The Alkaline Battery Adapter

An alkaline battery adapter is supplied with each instrument. The adapter (part number 059-3052-000) accepts four AA alkaline batteries (use only Duracell MN1500) and provides approximately 12 hours of operation. The adapter is intended to be used in emergency situations when there is no time to charge the Li-ion battery pack.

To insert batteries into the adapter:

- 1. Remove the three Philips-head screws to open the compartment.
- 2. Insert four fresh AA batteries as indicated by the polarity (+/-) markings.
- 3. Replace the cover. Replace the three screws.

To install the adapter in the instrument:

- 1. Remove the Li-ion battery pack from the battery compartment by sliding the tab and tilting out the battery.
- 2. Replace it with the alkaline battery adapter
- 3. Slide the tab back into place to secure the battery adapter.

IMPORTANT!

Alkaline batteries cannot be recharged. The instrument's internal circuit detects alkaline batteries and will not allow recharging. If you place the instrument in its cradle, the alkaline battery will not be recharged. The internal charging circuit is designed to prevent damage to alkaline batteries and the charging circuit when alkaline batteries are installed inside the instrument.

Note: When replacing alkaline batteries, dispose of old ones properly.

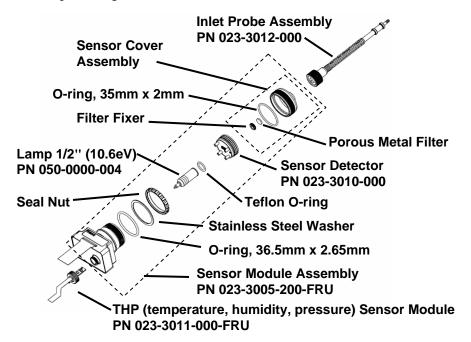
WARNING!

To reduce the risk of ignition of hazardous atmospheres, recharge the battery only in areas known to be non-hazardous. Remove and replace the battery only in areas known to be non-hazardous.

Note: The internal charging circuit is designed to prevent charging to alkaline batteries.

PID Sensor & Lamp Cleaning/Replacement

The sensor module is made of several components and is attached to the lamp-housing unit as shown below.



Sensor Components

Note: The cleaning procedure is not normally needed. Clean the PID sensor module, the lamp and the lamp housing only if:

- 1. The reading is inaccurate even after calibration.
- 2. The reading is very sensitive to air moisture.
- 3. A liquid has been sucked into the unit and damaged the unit.

Use of the external filter helps to prevent contamination of the sensor.

To access the sensor components and lamp, gently unscrew the lamphousing cap, remove the sensor adapter with the gas inlet probe and the metal filter all together. Then hold the PID sensor and pull it straight out. A slight, gentle rocking motion helps release the sensor.

Cleaning The PID Sensor

Place the entire PID sensor module into GC grade methanol. It is highly recommended that an ultrasound bath to be used to clean the sensor for at least 15 minutes. Then dry the sensor thoroughly. Never touch the electrodes of the sensor by hand.

Also use a methanol-soaked cotton swab to wipe off the lamp housing where it contacts the sensor when the sensor is installed.

Turn over the sensor so that the pins point up and the sensor cavity is visible. Examine the sensor electrodes for any corrosion, damage, or bending out of alignment. The metal sensor electrode "fingers" should be flat and straight. If necessary, carefully bend the sensor fingers to ensure that they do not touch the Teflon portions and that they are parallel to each other. Make sure that the nuts on the sensor pins are snug but not overtight. If the sensor is corroded or otherwise damaged, it should be replaced.

Cleaning The Lamp Housing Or Changing The Lamp

If the lamp does not turn on, the instrument will display an error message to indicate replacement of the lamp may be required.

1. If the lamp is operational, clean the lamp window surface and the lamp housing by wiping it with GC grade methanol using a cotton swab using moderate pressure. After cleaning, hold the lamp up to the light at an angle to detect any remaining film. Repeat the process until the lamp window is clean. Never use water solutions to clean the lamp. Dry the lamp and the lamp housing thoroughly after cleaning.

CAUTION: Never touch the window surface with the fingers or anything else that may leave a film. Never use acetone or aqueous solutions.

- 2. If the lamp does not turn on, remove the lamp from the lamp housing. Place the lamp O-ring onto the new lamp. Insert the new lamp, avoiding contact with the flat window surface.
- 3. Reinstall the PID sensor module.
- 4. Tighten the Lamp Housing Cap.

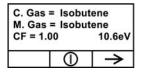
Determining The Lamp Type

The monitor can accommodate three lamp values: 10.6eV (standard), 9.8eV, and 11.7eV. The monitor automatically reads a marking on the side of the lamp to set the proper Correction Factor. There are two ways to determine the lamp type:

Remove the lamp and look for markings (bars) on the side:

No bars: 10.6eV1 bar: 11.7eV2 bars: 9.8eV

Also, when the monitor is running, the lamp type is shown along with the calibration and measurement gas and Correction Factor:



Note: This screen can be accessed from the reading screen by pressing [N/-] four times.

Sampling Pump

When approaching the end of the specified lifetime of the pump, it will consume higher amount of energy and reduce its sample draw capability significantly. When this occurs, it is necessary to replace or rebuild the pump. When checking the pump flow, make sure that the inlet connector is tight and the inlet tubing is in good condition. Connect a flow meter to the gas inlet probe. The flow rate should be above 450 cc/min when there is no air leakage.

If the pump is not working properly, refer the instrument to qualified service personnel for further testing and, if necessary, pump repair or replacement.

Cleaning The Instrument

Occasional cleaning with a soft cloth is recommended. Do not use detergents or chemicals.

Visually inspect the contacts at the base of the instrument, on the battery, and on the charging cradle to make sure they are clean. If they are not, wipe them with a soft, dry cloth. Never use solvents or cleaners.

Ordering Replacement Parts

If you need replacement parts, contact your local RAE Systems distributor. A list is available online:

http://www.raesystems.com

In the U.S., you can order sensors, replacement batteries, and other accessories online at:

http://istore.raesystems.com/

Special Servicing Note

If the instrument needs to be serviced, contact either:

1. The RAE Systems distributor from whom the instrument was purchased; they will return the instrument on your behalf.

or

2. The RAE Systems Technical Service Department. Before returning the instrument for service or repair, obtain a Returned Material Authorization (RMA) number for proper tracking of your equipment. This number needs to be on all documentation and posted on the outside of the box in which the instrument is returned for service or upgrade. Packages without RMA Numbers will be refused at the factory.

Troubleshooting

Problem	Possible Reas	ons & Solutions
Cannot turn on power	Reasons:	Discharged battery.
after charging the		Defective battery.
battery		
	Solutions:	Charge or replace battery.
Lost password	Solutions:	Call Technical Support at
		+1 408-752-0723 or toll-
		free at
		+1 888-723-4800
Reading abnormally	Reasons:	Dirty filter.
High		Dirty sensor module.
		Excessive moisture and
		water condensation.
		Incorrect calibration.
	Solutions:	Replace filter.
		Blow-dry the sensor
		module.
		Calibrate the unit.
Reading abnormally	Reasons:	Dirty filter.
Low		Dirty sensor module.
		Weak or dirty lamp.
		Incorrect calibration.
	Solutions:	Replace filter.
		Remove Calibration
		Adapter.
		Calibrate the unit.
		Check for air leakage.
Buzzer	Reasons:	Bad buzzer.
Inoperative		
	Solutions:	Check that buzzer is not
		turned off.
		Call authorized service
		center.

Inlet flow too low	Reasons:	Pump diaphragm damaged or has debris. Flow path leaks.
	Solutions:	Check flow path for leaks; sensor module O-ring, tube connectors, Teflon tube compression fitting. Call Technical Support at +1 408-752-0723 or toll-free at +1 888-723-4800
"Lamp" message	Reasons:	Lamp drive circuit.
during operation		Weak or defective PID
		lamp, defective.
	Solutions:	Turn the unit off and back on.
		Replace UV lamp

Technical Support

To contact RAE Systems Technical Support Team:

Monday through Friday, 7:00AM to 5:00PM Pacific (US) Time

Phone (toll-free): +1 888-723-4800

Phone: +1 408-952-8461

Email: tech@raesystems.com

Life-critical after-hours support is available:

+1 408-952-8200 select option 8

RAE Systems Contacts

RAE Systems World Headquarters

3775 N. First St.

San Jose, CA 95134-1708 USA

Phone: +1 408.952.8200 **Fax:** +1 408.952.8480

E-mail: customerserv@raesystems.com

Web Site: www.raesystems.com

RAE Systems Technical Support

Monday through Friday, 7:00AM to 5:00PM Pacific Time

Phone: +1.408.952.8461

Email: tech@raesystems.com

Life-critical after-hours support is available:

+1.408.952.8200 select option 9

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Phone: 81-3-5283-3268 **Fax:** 81-3-5283-3275

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Kyungki-Do, Korea **Phone:** 82-32-328-7123

Fax: 82-32-328-7127

Email: krsales@raesystems.com

Controlled Part of Manual

Intrinsic Safety:

US and Canada: Class I, Division 1, Groups A,B,C,D T4

Europe: ATEX (0575 Ex II 2G Ex ia IIC/IIB T4 Gb)

KEMA 07 ATEX 0127

Complies with EN60079-0:2009, EN60079-11:2007

IECEx CSA 10.0005 Ex ia IIC/IIB T4 Gb

Complies with IEC 60079-0:2007, IEC 60079-11:2006

Temperature: -20° C to 50° C (-4° to 122° F)

Humidity: 0% to 95% relative humidity (non-condensing)

Basic Operation

Turning The Instrument On

- 1. With the instrument turned off, press and hold [MODE].
- 2. When the display turns on, release the [MODE] key.

The instrument is now operating and performs self tests. Once the self tests are complete, the display shows a graph or numerical gas reading. This indicates that the instrument is fully functional and ready to use.

Turning The Instrument Off

- 1. Press and hold the Mode key for 3 seconds. A 5-second countdown to shutoff begins.
- 2. When you see "Unit off..." release your finger from the [MODE] key. The instrument is now off.

Note: You must hold your finger on the key for the entire shutoff process. If you remove your finger from the key during the countdown, the shutoff operation is canceled and the instrument continues normal operation.

Alarm Signals

During each measurement period, the gas concentration is compared with the programmed alarm limits (gas concentration alarm limit settings). If the concentration exceeds any of the preset limits, the loud buzzer and red flashing LED are activated immediately to warn you of the alarm condition.

In addition, the instrument alarms if one of the following conditions occurs: battery voltage falls below a preset voltage level, failure of the UV lamp, pump stall, or when the datalog memory is full.

Alarm Signal Summary

		1
Message	Condition	Alarm Signal
HIGH	Gas exceeds "High Alarm" limit	3 beeps/flashes per second*
OVR	Gas exceeds measurement range	3 beeps/flashes per second*
MAX	Gas exceeds electronics' maximum range	3 beeps/flashes per second*
LOW	Gas exceeds "Low Alarm" limit	2 beeps/flashes per second*
TWA	Gas exceeds "TWA" limit	1 Beep/flash per second*
STEL	Gas exceeds "STEL" limit	1 Beep/flash per second*
Pump icon flashes	Pump failure	3 beeps/flashes per second
Lamp	PID lamp failure	3 beeps/flashes per second plus "Lamp" message on display

Battery icon flashes	Low battery	1 flash, 1 beep per minute plus battery icon flashes on display
CAL	Calibration failed, or needs calibration	1 beep/flash per second
NEG	Gas reading measures less than number stored in calibration	1 beep/flash per second

Preset Alarm Limits & Calibration

The instrument is factory calibrated with standard calibration gas, and is programmed with default alarm limits.

Cal Gas (Isobutylene)	Cal Span	unit	Low	High	TWA	STEL
ppbRAE 3000	10	ppm	10	25	10	25
MiniRAE 3000	100	ppm	50	100	10	25
MiniRAE Lite	100	ppm	50	100	10	25
UltraRAE 3000	100	ppm	50	100	10	25

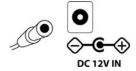
Charging The Battery

Always fully charge the battery before using the instrument. The instrument's Li-ion/NiMH battery is charged by placing the instrument in its cradle. Contacts on the bottom of the instrument meet the cradle's contacts, transferring power without other connections.

Note: Before setting the instrument into its charging cradle, visually inspect the contacts to make sure they are clean. If they are not, wipe them with a soft cloth. Do not use solvents or cleaners.

Follow this procedure to charge the instrument:

1. Plug the AC/DC adapter's barrel connector into the instrument's cradle.



- 2. Plug the AC/DC adapter into the wall outlet.
- 3. Place the instrument into the cradle, press down, and lean it back. It locks in place and the LED in the cradle glows.

Note: To release the instrument, press down and tilt the top out of the cradle and lift up.

The instrument begins charging automatically. The LED on the front of the cradle marked "Primary" blinks during charging. During charging, the diagonal lines in the battery icon on the instrument's display are animated and you see the message "Charging..."

When the instrument's battery is fully charged, the battery icon is no longer animated and shows a full battery. The message "Fully charged!" is shown and the Primary LED on the cradle glows continuously green.

Note: A spare Li-ion battery (059-3051-000) or NiMH(059-3054-000) can be charged by placing it directly in the charging port on the back of the cradle. It can be charged at the same time as the instrument. Press the battery in place, sliding it slightly toward the front of the cradle. This locks it in the cradle. To release the battery, slide it forward again and tilt it up.

Note: An Alkaline Battery Adapter (part number 059-3052-000), which uses four AA alkaline batteries (Duracell MN1500), may be substituted for the Li-Ion battery.

WARNING!

To reduce the risk of ignition of hazardous atmospheres, recharge and replace batteries only in areas known to be non-hazardous. Remove and replace batteries only in areas known to be nonhazardous.

Low Voltage Warning

When the battery's charge falls below a preset voltage, the instrument warns you by beeping once and flashing once every minute, and the battery icon blinks once per second. You should turn off the instrument within 10 minutes and either recharge the battery by placing the instrument in its cradle, or replace the battery with a fresh one with a full charge.

Clock Battery

An internal clock battery is mounted on one of the instrument's printed circuit boards. This long-life battery keeps settings in memory from being lost whenever the Li-ion, NiMH, or alkaline batteries are removed. This backup battery should last approximately five years, and must be replaced by an authorized RAE Systems service technician. It is not user-replaceable.

WARNING

To reduce the risk of ignition of hazardous atmospheres, recharge battery only in area known to be non-hazardous. Remove and replace battery only in an area known to be non-hazardous.

Replacing Rechargeable Li-Ion or NiMH Battery

Caution: Turn off the instrument before removing or replacing the battery.

Alkaline Battery Adapter

An alkaline battery adapter is supplied with each instrument. The adapter (part number 059-3052-000) accepts four AA alkaline batteries (use only Duracell MN1500).

Do not mix old and new batteries or different type batteries.

Troubleshooting

Problem	Possible Reas	ons & Solutions
Cannot turn on power	Reasons:	Discharged battery.
after charging the		Defective battery.
battery		
	Solutions:	Charge or replace battery.
Lost password	Solutions:	Call Technical Support at
		+1 408-752-0723 or toll-
		free at
		+1 888-723-4800
Reading abnormally	Reasons:	Dirty filter.
High		Dirty sensor module.
		Excessive moisture and
		water condensation.
		Incorrect calibration.
	Solutions:	Replace filter.
		Blow-dry the sensor
		module.
		Calibrate the unit.
Reading abnormally	Reasons:	Dirty filter.
Low		Dirty sensor module.
		Weak or dirty lamp.
		Incorrect calibration.
		5 1 01
	Solutions:	Replace filter.
		Remove Calibration
		Adapter.
		Calibrate the unit.
	_	Check for air leakage.
Buzzer	Reasons:	Bad buzzer.
Inoperative	G 1 4	
	Solutions:	Check that buzzer is not
		turned off.
		Call authorized service
		center.

Inlet flow too low	Reasons:	Pump diaphragm damaged or has debris. Flow path leaks.
	Solutions:	Check flow path for leaks; sensor module O-ring, tube connectors, Teflon tube compression fitting. Call Technical Support at +1 408-752-0723 or toll-free at +1 888-723-4800
"Lamp" message	Reasons:	Lamp drive circuit.
during operation		Weak or defective PID
		lamp, defective.
	Solutions:	Turn the unit off and back
		on.
		Replace UV lamp



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> Rev. C August 2010 P/N 059-4020-000

APPENDIX C Field Data Collection Forms



Boring Log Clie		Project Client:				Log of B	oring		
				Project	Number:				
Date(s) Drilled Drilling					Logged By Diameter of		Checked By	Total Depth of Borehole (ft) Ground Surface	Depth to Water (bgs)
Method					Borehole (in)		Elevation (ft-msl)	
Drill Rig					Drilling			Groundwater Elevation (ft-msl)	
Type Driller's Na	me		Sampler Type		Company			Measuring Point Elevation (ft-msl)	
Descr	iption (ole Locat	ion				Northing Easting	
	SA	MPL	ES						
Depth (ft-bgs)	Blows/6"	Recovery (ft)	PID (ppm)	USCS Symbol	Graphic Log	MATE	RIAL DESCR	RIPTION	REMARKS
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Boring Log			Project Client: Project	Name: Number:		Log of B	oring	
	SA	MPL	ES					
Depth (ft-bgs)	Blows/Foot	Recovery (ft)	PID (ppm)	USCS Symbol	Graphic Log	MATERIAL DESCRIP	PTION	REMARKS
8								

ATTACHMENT 1 Site Safety and Health Plan



FINAL SITE SAFETY AND HEALTH PLAN

PERFORMANCE BASE REMEDIATION TASK ORDER FOR PETERSON AIR FORCE BASE SITES OW011 and OW012

Contract No: FA8903-09-D-8578

Task Order: 0003

SubCLINs: 0009EV and 0009EX

February 2013



Air Force Civil Engineer Center 2261 Hughes Avenue, Suite 155 Lackland AFB, Texas 78236-9853

Prepared by



RMA-Insight Joint Venture 3061 East La Palma Avenue Anaheim, California 92806

in association with







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Attachment A Activity Hazard Analyses

LIST OF ACRONYMS AND ABBREVIATIONS

mg/kg milligrams per kilogram mg/m³ milligrams per cubic meter

AFB air force base

AFCEC Air Force Civil Engineer Center

AHA Activity Hazard Analysis
CFR Code of Federal Regulations

COC chemicals of concern CO carbon monoxide

COR Contract Officer's Representative CRZ Contamination Reduction Zone

CSEV-R Colorado Soil Evaluation Values - Residential

dBA Decibel, A weighted

DERA Defense Environmental Remediation Account

DFW Definable feature of work

EPA United States Environmental Protection Agency

ESQ Environment, Safety, Quality

EZ Exclusion Zone

ft feet

HAZWOPER Hazardous Waste Operations and Emergency Response

IRP Installation Remediation Program

LEL lower explosive limit

NA Not applicable

 O^2 oxygen

OSHA Occupational Safety and Health Administration

OWS Oil/water separator

PAH Polyaromatic Hydrocarbons
PEL Permissible Exposure Limit
PPE Personal Protective Equipment

PPM parts per million RI remedial investigation

SOP Standard Operating Procedure SSHO Site Safety and Health Officer SSHP Site Safety and Health Plan

SZ Support Zone TCE trichloroethene

TLV Threshold Limit Value

TPH-DRO total petroleum hydrocarbon– diesel range organics
TPH-GRO total petroleum hydrocarbon–gasoline range organics

USACE US Army Corps of Engineers

USAF US Air Force

VOC volatile organic compounds WBGT Wet Bulb Globe Temperature



Jon Vail

Project Manager

1.0 INTRODUCTION AND SIGNATURES

1 1

This Site Safety and Health Plan (SSHP) has been prepared by ECC for RMA-Insight Joint Venture to conduct soil sampling and assessments at Peterson Air Force Base (AFB), located in Colorado Springs, Colorado. The SSHP presents health and safety information, procedures, and policies necessary for the safe implementation of work tasks. ECC is the primary subcontractor for this site for work efforts and task implementation, under contract to RMA-Insight. All activities will be coordinated with and through RMA-Insight. Onsite activities are regulated by the United States Environmental Protection Agency (EPA) and the Colorado Department of Public Health and the Environment. Work conducted under this contract will be performed in accordance with applicable Federal, state, and local safety and occupational health laws and regulations including: Occupational Safety and Health Administration (OSHA) standards (including 29 Code of Federal Regulations [CFR] 1910 and 29 CFR 1926) and the United States Army Corps of Engineers (USACE) Safety and Health Requirements Manual (EM 385-1-1, 15 November 2008, as updated). Because this remediation effort is governed by the National Oil and Hazardous Substances Pollution Contingency Plan, the requirements of the Hazardous Waste Operations and Emergency Response (HAZWOPER) will apply. The contents of the SSHP are subject to review and revision as new information becomes available.

Additional safety and health requirements are found in Activity Hazard Analyses (AHAs) (Attachment A) and Environment Safety and Quality (ESQ) standard operating procedures (SOP) as identified in this plan.

Plan Preparer:		
Mo manage	02/14/2013	3
		303-298-7607
John Ryder	Date	Phone Number
Assistant Project Manager		
Plan Reviewer: M Johnshoy CIH, C	Marcus Johnshoy 2013.02.14 14:23:26 -06'00'	8
	-06'00'	303-887-7427 cell
Marcus Johnshoy, CIH, CSP Regional ESQ Manager	Date	Phone Number
Plan Approval:	/7	

2.0 PROJECT/TASK ORDER DESCRIPTION

2.1 Site Description

Peterson AFB is located on the eastern border of Colorado Springs, El Paso County, Colorado, and provides runways for the adjacent City of Colorado Springs Municipal Airport under a shared joint civil-military airport agreement. The installation is approximately 1,278 acres in size and is at an elevation of 6,035 feet above mean sea level.

2.2 Site History

2.2.1 Description and Previous Investigation of Site OW011

Site OW011 at Peterson AFB consists of an abandoned concrete oil/water separator (OWS) (approximately 4 feet [ft] by 4 ft by 9.5 ft deep) that previously received floor wash water from floor drains; the water may have contained petroleum, oil, and lubricants. A 2008 report noted the vault beneath the sewer covers had been filled in with concrete to a depth of approximately 4 inches below ground surface; however, no information regarding actions taken to prevent discharges to and from the OWS was available.

Soil samples were collected in April 11, 2011, during an investigation of the OWS to determine Defense Environmental Remediation Account (DERA) eligibility. Soil samples collected from the anticipated OW011 influent pipe line were selected for laboratory analyses based on high photoionization detector readings and were analyzed by an off-site laboratory for volatile organic compounds (VOCs), total petroleum hydrocarbon—gasoline range organics (TPH-GRO), and TPH-diesel range organics (TPH-DRO). The April 2011 DERA Investigation Report concluded that the one soil sample identified a potential release from the OWS, based on the TPH-DRO concentration of 2,400 milligrams per kilogram (mg/kg); therefore, the other two samples were not analyzed, consistent with the DERA protocols. However, the other detected analytes at Site OW011 did not exceed the residential Colorado Soil Evaluation Values (CSEV-R).

2.2.2 Description and Previous Investigation of Site OW012

Site OW012 at Peterson AFB includes an area near Hole 2 at the Peterson AFB Golf Course. The site is within the boundaries of a former Installation Remediation Program (IRP) site that contained a leach field system. The former leach field consisted of a settling tank (likely the OWS for Site OW012) and a gravel-envelope drainage field. A Remedial Investigation (RI) for the former IRP site was completed in 1989. Data and information collected during the RI demonstrated no adverse impact to human health or the environment, based on a risk assessment, and it was determined that no further action was necessary. Based on those findings, the site was eliminated from further consideration under the IRP.

A 2008 report indicated magnetic anomalies were observed at two locations approximately 140 ft south of Hole 2 and 30 ft east of a north/south golf cart road. The April 2011 DERA investigation sampling locations were based on this information and personnel interviews indicated that a formerly active OWS may be located in this area. The estimated location of Site OW012 is within the footprint of the leach field settling tank as depicted in the 1989 RI report. For the 2011 DERA investigation, three borings were drilled approximately 50 ft apart around Site OW012 (based on the magnetic anomalies), and one sample was collected from each boring.

The only sample analyzed at the laboratory was collected approximately 100 ft east of the OWS; naphthalene and trichloroethene (TCE) were detected above CSEV-R in this sample.

Site locations and previous sampling locations are presented in Figures 2-1, 2-2, and 2-3.

2.3 Scope of Work

The objectives of the sampling efforts at the OWS sites are two-fold:

- 1. Collect sufficient data to delineate the vertical and horizontal extent of contamination for possible excavation and disposal.
- 2. Collect sufficient soil data to allow development of Exposure Point Concentrations for each OWS Site and establish comparisons to the Colorado Soil Evaluation Values

2.4 Definable Features of Work

The Definable Features of Work (DFW) identified for the Site are provided in Table 2-1 below:

Table 2-1. Definable Features of Work for OWS Sites Peterson AFB, Colorado Springs, CO

1 0001201111129,	0020200
Mobilization and Site Preparation	

Collection of soil samples – hand sampling of surface soil to 3 inches; direct push sampling of subsurface soil to 15 feet.

Coring through concrete and asphalt cover to access subsurface soil locations in areas with existing cover material in place.

2.5 Key Project Personnel

Table 2-2 lists key personnel for this project.

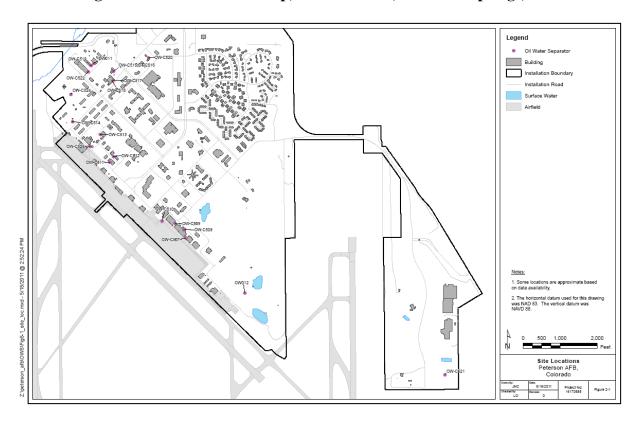
Table 2-2. Key Personnel Peterson AFB, Colorado Springs, CO

Name	Title	Organization	Phone Number/email
Ernesto Perez	Contract Officer's Representative; Project Manager	Air Force Civil Engineer Center (AFCEC)	307-773-3468 Ernesto.perez@us.af.mil
Robert Fant	Chief, Environmental Quality	21 CES/CEAN	719-556-6100 Robert.fant.1@us.af.mil
Sharon Stone	ERA Project Manager	HQ AFSPS/A7M	719-554-5819 Sharon.stone@us.af.mil
Mitra Fattahipour	Senior Project Manager	RMA-Insight	858-342-5585 mfattahipour@ieeci.com
David Cox	Project Manager/Denver	RMA-Insight	720-250-8551
Nick Weinberger	Quality Assurance Manager	RMA-Insight	714-678-6700
Maureen Sassoon	Health and Safety Manager	RMA-Insight	909-782-8545
John Ryder	Project Manager	ECC	720-232-6425 jryder@ecc.net

Table 2-2. Key Personnel Peterson AFB, Colorado Springs, CO

Name	Title	Organization	Phone Number/email
Marcus	Site Safety and Health	ECC	303-887-7427
Johnshoy	Officer	ECC	mjohnshoy@ecc.net

Figure 2-1. Site Location Map, Peterson AFB, Colorado Springs, CO



2-3

Figure 2-2. Site OW011 – Previous Sampling Locations

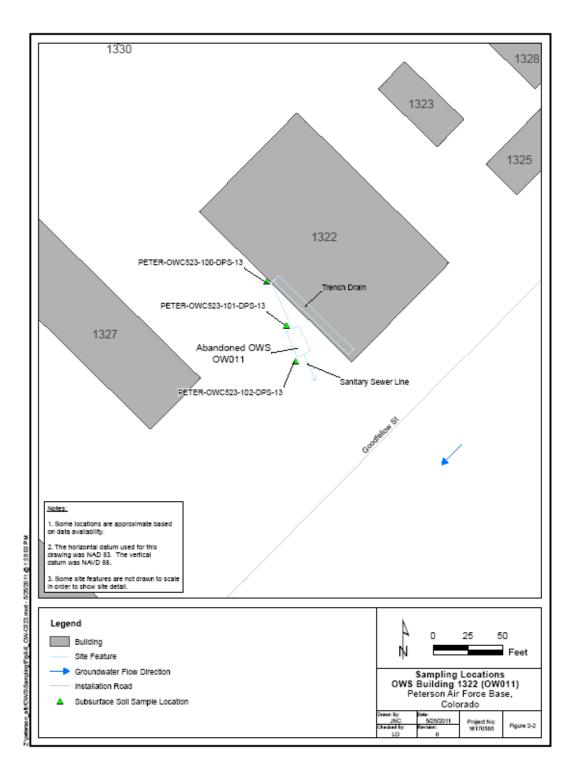
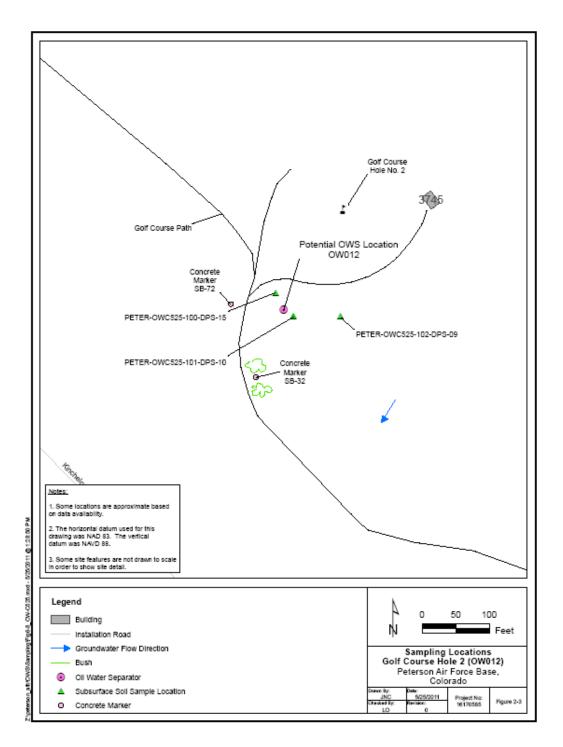


Figure 2-3. Site OW012 – Previous Sampling Locations



3.0 HAZARD ASSESSMENT

Project hazards will be controlled by following established Standard Operating Procedures (SOP) and Activity Hazard Analysis (AHAs), as identified in Table 3-1. AHAs for field activities are included in Attachment A of this SSHP. The AHAs shall be reviewed with work crew before commencing any work and revised throughout the project, as necessary. If hazards or conditions are identified that are not covered by this SSHP, the ECC Project Manager and the project Site Safety and Health Officer (SSHO) must be contacted ECC project staff must contact the Project Manager and the SSHO for hazard evaluation and appropriate control methods.

Note that additional AHAs will be prepared for work tasks not addressed here. These new AHAs will be appended to this SSHP.

Table 3-1. Referenced SOPs and AHAs Peterson AFB, Colorado Springs, CO

Referenced SOPs and AHAs:			
ECC SOPs applicable to this project	TOOLS WITH THE TAIL		
SOP ESQ-1.8 Drug Free Workplace	SOP ESQ-8.3 Bloodborne Pathogens		
SOP ESQ-2.2 Incident Reporting and	SOP ESQ-8.5 Heat Stress Prevention		
SOP ESQ-5.3 Mobile Construction	SOP ESQ-8.6 Hearing Conservation		
SOP ESQ-5.4 Motor Vehicle Operations	SOP ESQ-8.7 Air Monitoring		
SOP ESQ-5.7 Electrical Safety	SOP ESQ-8.8 Biological Hazards		
SOP ESQ-5.8 Hand and Power Tools	SOP ESQ-9.3 Waste Management		
SOP ESQ-5.9 Fire Protection	SOP HS-025 Emergency Response		
SOP ESQ-5.10 Hazard Communication	SOP ENV-301 Equipment Inspection		
SOP ESQ-5.13 Back Injury Prevention	SOP ENV-302 Equipment Decontamination		
SOP ESQ-6.1 Personal Protective	SOP ENV-303 Heavy Equipment		
SOP ESQ-7.6 Underground Utilities	SOP ENV-603 Field Documentation		
SOP ESQ-8.1 Contamination Control	SOP 3-16 Downhole Boring and Sampling		
SOP ESQ-8.2 Medical Surveillance			
AHAs			
Mobilization and Site Preparation	Direct Push Drilling and Soil Sampling		

3.1 Chemical Hazards

In soil, the primary chemicals of concern (COCs) are VOCs and semi-volatile organic compounds. Because of the limited volatility of these chemicals, dermal contact is the most significant route of exposure. Table 3-2 provides chemical hazard information for these COCs in soil. The majority of detections for these chemicals range from slightly above the Colorado residential cleanup standard to approximately four times the standard.

In accordance with EM 385-1-1, exposure to any chemical, biological, or physical agent is prohibited in excess of the acceptable limits specified in the most recent American Conference of Industrial Hygienists guidelines or by OSHA, whichever is more stringent. In case of conflicts between standards and regulations, the more stringent exposure limit shall prevail.

Table 3-2. Chemical Hazard Information for OWS Sites Peterson AFB, Colorado Springs, CO

Chemical of	Maximum Site	Exposure	Route of Entry	Target Organs
Concern	Concentration	Limits	Exposure Symptoms	Turget Organis
Concern	(mg/kg)	Ziiiits	Exposure symptoms	
TCE	0.18	PEL: 100 ppm TLV: 10 ppm	Inhalation, ingestion, skin and/or eye contact • Irritation of eyes and skin, headache, visual disturbance,	Kidneys, liver, eyes, skin, central nervous system, cardiovascular system, reproductive
			weakness, dizziness, tremors, drowsiness, nausea, dermatitis; cardiac arrhythmias; liver injury; potential male reproductive toxin. Potential carcinogen.	system
Naphthalene	45	PEL: 10 ppm	Inhalation, ingestion, skin and/or eye	Eyes, respiratory
Tupitulaielle		1 EE 10 pp	contact	system, liver, kidneys,
Disable of	2.400	TLV: 10 ppm	Eye irritation; headache; confusion, excitement; malaise; nausea, vomiting, abdominal pain; bladder irritation; profuse sweating; jaundice; hematuria (blood in the urine; dark urine), renal shutdown; dermatitis, optical neuritis, corneal damage; cataracts.	central nervous system, gastrointestinal tract
Diesel Fuel	2,400	PEL:	Inhalation, skin absorption, ingestion, skin and/or eye contact • Eye irritation, dermatitis,	Eyes, respiratory system, kidneys, central nervous system, lungs,
		mg/m ³	respiratory tract irritation, dizziness. Potential carcinogen.	gastrointestinal tract
PAHs *	NA	PEL: 0.2	Inhalation, ingestion, skin and/or eye contact	Respiratory system, skin, bladder, kidneys.
		TLV: 0.2 (Both as coal tar pitch volatiles)	Dermatitis, bronchitis, carcinogen	Lung, kidney, and skin cancer.

mg/m³ – milligrams per cubic meter

PAH – Polyaromatic Hydrocarbons

PEL – permissible exposure limit

TVL - Threshold Limit Values

Table 3-3. Identification of Chemicals of Concern for OWS Sites

Peterson AFB, Colorado Springs, CO

SITE	PAHs	TCE	Diesel Fuel	Naphthalene
OW011	X		X	
OW012	X	X		X

^{*} PAHs were not analyzed for in previous investigations

4.0 PERSONAL PROTECTIVE EQUIPMENT

4.1 Selection

Unless otherwise approved by the SSHO and documented in an AHA, all activities will include, at minimum, use of American National Standards Institute approved hard hats, safety-toe footwear and safety glasses with side impact protection. Hard hats are required when there is a risk of head injury from overhead hazards. High visibility garments must be worn whenever workers are exposed to vehicular or heavy equipment traffic and by all ground personnel when supplemental lighting is used in work areas during periods of insufficient daylight. Table 4-1 summarizes the personal protective equipment (PPE) anticipated during the planned field activities.

Table 4-1. PPE Selection Peterson AFB, Colorado Springs, CO

Activity	Respiratory Protection *	Body Protection	Hand Protection	Eye/Face Protection	Hearing Protection
Mobilization, Site Inspection, Site Preparation	NA	Level D	Leather work gloves as necessary	Safety glasses	Plugs or muffs as necessary to reduce exposure to <85dBA
Direct push soil sampling	NA	Level D	Leather work gloves; latex/nitrile gloves**	Safety glasses	Plugs or muffs as necessary to reduce exposure to <85dBA

dBA – decibels

4.2 Use and Limitations

Read and follow all manufacturer instructions regarding product use and limitations.

Hand protection – Use gloves as described in Table 4-1. Nitrile or latex protective gloves are meant for incidental contact with contaminated soil. Inspect frequently and change gloves on signs of contamination, and when protection is compromised by wear and tear. Nitrile/latex gloves will be changed at each sample collection location and discarded properly. Wash hands thoroughly before eating or smoking.

Eye/face protection – Wear protection prescribed in Table 4-1. Replace safety glasses when scratched or damaged.

Hearing Protection – Wear hearing protective devices in accordance with manufacturers' instructions, including fitting and insertion. Hearing protection is required whenever task operations results in noise exposures in excess of 85 dBA slow.

NA – not applicable

^{*} Use of respiratory protection during project activities is not anticipated to be required based on the contaminants of concern and their concentrations in soil.

^{**} Latex/nitrile gloves as necessary to prevent contact with contaminated media during work task.

4.3 PPE Maintenance and Storage

It is important that all PPE be kept clean and properly maintained. Cleaning is particularly important for eye, face, and respiratory protection where dirty or fogged lenses could impair vision. Employees must inspect, clean, and maintain their PPE according to the manufacturers' instructions before and after each use. Supervisors are responsible for ensuring that users properly maintain their PPE in good condition.

Personal protective equipment must not be shared between employees until it has been properly cleaned and sanitized. PPE will be distributed for individual use whenever possible. Where employees provide their own protective equipment, ECC will be responsible to assure its adequacy, including proper maintenance, and sanitation of such equipment.

4.4 PPE Decontamination and Disposal

It is anticipated that most of the PPE utilized on the project, beyond the basic PPE elements (hard hat, safety glasses, steel-toed boots, leather gloves), will be of a disposable nature (nitrile or other chemical gloves, ear plugs). Contaminated PPE will be collected in a dedicated container at the contamination reduction zone and disposed of appropriately at a suitable facility. Based on the contaminants present and their concentrations, it is anticipated that disposable PPE will be disposed of as non-hazardous waste.

Ear muffs, safety glasses, and other non-disposable forms of PPE will be routinely wiped clean of any visible contamination after each use.

4.5 PPE Training and Proper Fitting

Users will be trained on the selection and use and limitations of PPE. Each affected employee will demonstrate an understanding of the training specified in this section, and the ability to use PPE properly, before being allowed to perform work requiring the use of PPE.

When ECC has reason to believe that any affected employee, who has already been trained, does not have the understanding and skill required of this section, the employee will be re-trained.

Gloves, head gear, and footwear must fit properly to avoid tripping and snagging hazards or constriction and tearing. Hearing protection will be checked by the user and SSHO for proper fit and must be inserted/worn in accordance with manufacturer instructions.

4.6 PPE Donning and Doffing Procedures

The SSHO will train employees and demonstrate proper donning and doffing procedures for PPE ensembles worn in exclusion zones during site orientation.

4.7 PPE Inspection Procedures

Each person who is required to wear PPE will inspect their equipment prior to, during, and after each use. Defective or damaged equipment will not be used. Heavily contaminated PPE that cannot be adequately cleaned will be discarded. Stored PPE will be inspected as needed to ensure that it has not been damaged and is suitable for use. Workers who wear PPE in the field will take note of the condition of the PPE worn by their co-workers and inform them of any apparent problems, such as, missing equipment, tears in protective clothing, excessive contamination, improper use of PPE, and inadequate PPE.

4.8 Evaluation of Effectiveness of PPE Program

The SSHO will inspect the jobsite each day and as frequently as necessary to ensure that PPE has been properly selected and is being used as designed. The SSHO will also inspect PPE stored for emergency use.

5.0 MEDICAL SURVEILLANCE

Employees on this project whose work involves potential exposure to site contaminants will be included in the medical surveillance program.

5.1 General

All field personnel will have a current medical exam clearance (physician's written opinion) in accordance with OSHA standards prior to entering regulated work areas in accordance with 29 Code of Federal Regulations 1910.120 in addition to the required HAZWOPER training. ECC utilizes the services of WorkCare, whose principal is Dr. Peter Greaney, a Board Certified Occupational Physician, to review all exams and provide the clearances, which include an assessment of the employee's ability to use respiratory protection and PPE safely. The SSHO will ensure that subcontractor employee medical screenings are completed, documented, and available for inspection.

5.2 Special Medical Exams and Biological Monitoring

Physiological monitoring for heat stress may be performed in accordance with SOP ESQ-8.5 (Heat Stress Prevention).

6.0 HEALTH AND HAZARD MONITORING

6.1 Real Time Air Monitoring

Table 6-1 below lists the monitoring requirements and response actions for this project. All direct and integrated air monitoring equipment will be properly calibrated before and after each period of use in accordance with the manufactures' instructions and standard industrial hygiene practice.

ECC's monitoring program will be performed by trained personnel who are knowledgeable in calibration of the equipment and interpretation of results. Personnel performing testing and monitoring will be trained in testing and monitoring procedures and hazards. Monitoring equipment will be used, inspected, maintained, and properly calibrated in accordance with the manufactures' instructions and standard industrial hygiene practice.

Table 6-1: Health and Hazard Monitoring Peterson AFB, Colorado Springs, CO

Peterson Arb, Colorado Springs, CO					
	Real Time	(Air, noise, heat, dust)			
Instrument / Contaminant	Frequency	Action Levels	Actions/Upgrade and Rationale		
Multi-gas Meter / O²/LEL/H ₂ S/CO	Not planned during intrusive these constituents	activities; no expectation	n of hazardous atmospheres related to		
Photoionization Detector / VOCs	During direct push soil sampling, sufficient to characterize potential exposures for subsequent sampling events.	5 ppm in the breathing zone	Move upwind, allow for concentrations to dissipate. If concentrations do not decrease, conduct further assessment of hazards and controls before proceeding with task.		
Noise Monitoring	Prior to the start of noise generating activities	85 dBA	Implement hearing conservation program & utilize appropriate hearing protection PPE		
Heat Stress (WBGT) + Body Tem / Heart Rate Monitoring	As required by ECC SOP 8.5 based on work activity, PPE usage, and ambient temperature	Variable depending on the individual and work activity	Participate in heat stress monitoring program, take breaks in the shade, drink fluids		
Dust Monitor	Not planned during intrusive	activities; no expectation	n of excessive dust generation		
		sonal Exposure Monito			
Contaminant	Estimated Frequenc		Duration / Actions & Upgrade		
	Based on the concentrations of contaminants in the soil, personal exposure monitoring for exposure to contaminants is not expected to be necessary.				

Notes:

CO – carbon monoxide

H₂S - Hydrogen sulfide

LEL – lower explosive limit

 O^2 - oxygen

ppm – parts per million

WBGT – Wet Bulb Globe Temperature

7.0 CONTAMINATION CONTROL, SANITATION AND WASTE MANAGEMENT

The following sections describe the contamination control during the field activities.

7.1 Work Zones

Access to the work areas will be limited to authorized project personnel, approved subcontractors and their employees, Air Force and Lockheed Martin representatives, and local/State/Federal agency staff escorted by site personnel. All personnel must provide proof of HAZWOPER training before entering a delineated work area. Specific work areas will be established and controlled as defined below.

7.1.1 Support Zone

The Support Zone (SZ) is located outside the Contamination Reduction Zone (CRZ). The support zone is an uncontaminated (clean) area where personnel will not be exposed to hazardous materials or contaminants. Additional supplies are located here for use as needed – e.g., PPE, drinking water, tools, additional decontamination supplies, fire extinguishers.

7.1.2 Contamination Reduction Zone

The CRZ is located in between and serves as a buffer between the SZ and the Exclusion Zone (EZ). The CRZ is intended to prevent the spread of contamination and is utilized for both personnel and equipment decontamination. Used PPE will be discarded properly in a labeled container. An entry log will be maintained at the CRZ with personnel names and in/out times for work in the EZ.

7.1.3 Exclusion Zone

The EZ is the work area where there is potential for worker exposure to site contaminants. The EZ is accessed only through CRZ. The exclusion zone perimeter will be delineated with caution tape, traffic cones, or other high visibility methods.

7.2 Decontamination Procedures

Heavy machinery and vehicles

Dry decontamination with shovels, malets, etc. to remove gross contamination and then broom or brush swept to a clean surface condition. Wet decontamination procedures will be utilized on drilling equipment to reduce the potential for cross contamination between sites. All decontamination liquids will be captured, contained, and properly disposed.

Hand Tools, Power Tools, and other Tools/Supplies:

Remove gross contamination by appropriate means and then broom or brush swept to a clean surface condition. All soil sampling equipment and tools will be properly decontaminated between locations.

Personnel:

The importance of good hygiene practices will be emphasized with all site workers. Upon exiting the EZ and CRZ, workers and/or site visitors will enter the CRZ and carefully remove all designated PPE, wash their hands, face, and forearms prior to eating, drinking or smoking. Smoking, eating and drinking will only be allowed in clean, designated work areas (i.e., SZ).

7.3 Additional Special Work Practices

Dust control is not expected to be a concern during drilling and sampling activities. If excessive dust is generated, work activities will be altered to reduce dust generation, or a water spray will be utilized to control dust.

The use of the buddy system will be practiced during all site activities. Under no circumstances will a worker be allowed to perform field work activities without a partner.

7.4 Sanitation

Drinking water will be provided for work crews. Any project-related water outlets dispensing non-potable water will be conspicuously posted "Caution – Water Unsafe for Drinking, Washing, or Cooking."

If existing toilet facilities at Peterson AFB are not available for project use, portable toilet facilities will be provided for site work. Waterless hand sanitizer will be provided at each facility. Portable toilet facilities shall be periodically serviced by commercial vendor at a frequency necessary to maintain satisfactory conditions.

Separate shower facilities for this project are not required by 29 CFR 1926.65 (HAZWOPER).

Project waste and general trash will be collected. An appropriate number of waste receptacles will be available on the job site. Lids on these receptacles shall be of a locking fashion, and containers shall be secured as necessary to prevent tip over and loss of debris. Trash shall be removed from the site daily or as necessary and disposed of as normal nonhazardous waste. Used PPE and sampling supplies can be disposed of as routine nonhazardous waste.

8.0 EMERGENCY PLANNING AND REPORTING

8.1 Hospital and Emergency Route

Table 8-1 and Figure 8-1 provide phone numbers for emergency notification and a hospital route map. Local hospital emergency rooms must be notified of the potential types of injuries and the contaminants involved. All personnel must be informed of emergency procedures and phone numbers during project orientation. Site vehicles must keep a copy of the hospital route map and emergency phone numbers in project vehicles at all times.

8.2 Emergency Supplies

At a minimum, the following supplies/equipment will be immediately available at each work location:

- First aid equipment and supplies;
- Blood borne pathogen response supplies;
- Spill control material and equipment; and
- Type ABC fire extinguisher, (2A:10BC, minimum of two).

8.3 Training

All personnel will be trained to the emergency procedures and phone numbers. At least two individuals will be onsite with current First Aid/cardiopulmonary resuscitation certification.

8.4 Incident Investigation, Reports, Logs

All incidents are to be reported immediately to the ECC Project Manager and the SSHO. Incidents will include:

- Injuries requiring only first aid treatment;
- OSHA-recordable injuries or illnesses (e.g., medical treatment beyond first aid);
- Injuries to authorized visitors or the general public;
- Fires and explosions of any magnitude;
- Spills and environmental releases;
- Tool or equipment failure which results (or could result in) serious injury;
- Property damage, equipment damage, or environmental damage resulting in a loss of more than \$500 (If \$2,000 or more it will be reported to AFCEC); and
- Any event which, under slightly different circumstances, could have resulted in one of the above.

All injuries, illnesses, and property damage accidents will be reported to AFCEC Contract Officer's Representative (COR) as soon as possible, but no later than 24 hours after the incident. Investigation findings and corrective actions will be reported within 5 days following an accident. The supervisor, with the assistance of the SSHO, will investigate the incident and complete all necessary incident reports and logs, including the ECC Incident Report and AFCEC or regulatory agency reports. All incidents, regardless of severity, require some type of

investigation and corrective action. Immediate and basic causes will be identified, evaluated, and used to support the recommended corrective actions.

8.5 Immediate Notification of Major Accidents

The COR will be verbally notified immediately of any incidents that involve, or appear to involve:

- A fatal injury;
- A permanent total disability;
- A permanent partial disability;
- The hospitalization of three or more people resulting from a single occurrence;
- Property damage of \$2,000 or more;
- An arc-flash incident/accident;
- A weight-handling mishap; and
- A High Visibility Accident (may generate publicity or high visibility).

At the time of any major incident, project site conditions will be preserved until released by the Government investigation team.

The written report will be submitted to AFCEC no later than 5 working days after the accident. Corrective actions will be implemented as soon as possible.

8.6 Severe Weather

On site operations will be discontinued during heavy precipitation or periods of strong or gusty winds or other severe weather. All outdoor activities will cease and employees will seek shelter during any periods of severe weather warning (e.g., severe thunderstorms, tornadoes, etc.). In the event of a tornado warning, project personnel should proceed immediately to the interior or basement of a substantial Peterson AFB building. If there is no time to seek shelter, go to a low-lying area away from any debris and cover your head. No site operations will be permitted during thunderstorms. When a thunderstorm approaches the site (e.g., thunder within 8 seconds after lightning), personnel will be evacuated from locations that present lightning hazards or all personnel must move inside site vehicles for cover until an all clear is given by the SSHO. If high winds are present at the site, the following actions will be taken:

If high winds exceeding 35 mph are present at the site, the following actions will be taken:

- All materials and equipment will be secured.
- All personnel will seek local shelter.
- All equipment will be protected from potential damage or from damaging the facility property.
- All equipment will be sheltered from exposure to excessive rain (if applicable).

When there is an imminent threat of electrical storms, the following actions will be taken:

- Seek shelter in a vehicle with windows rolled up and doors closed, or in a safe building.
- Avoid high elevations or elevated objects (trees, towers, etc.)

- Allow the storm to pass.
- The SSHO will assess site conditions and damage after the storm and prior to resumption of work.
- Work will not resume until 30 minutes has passed since the last lightning is observed or thunder is heard

Table 8-1. Emergency Contact List Peterson AFB, Colorado Springs, CO

Em	Emergency and POC Phone Numbers				
Emergency:	911				
Fire, Medical,					
Security, Spill					
Fire - Nonemergency	(719) 554-5112				
Security - Nonemergency	(719) 556-4000				
Memorial Hospital Central	(719) 365-5000				
AFCEC COR, Project Manager (Ernesto Perez)	(307) 773-3468 (office)				
ECC Project Manager (John Ryder)	(720) 232-6425 (cell)				
ECC Site Safety and Health Officer (Marcus Johnshoy)	(303) 887-7427 (cell)				
EPA (24-Hour Emergency)	(303) 294-7142				
Poison Control Center	(800) 442-2702				

KEY PROJECT PERSONNEL ARE TO HAVE A COPY OF THIS LIST READILY AVAILABLE AT ALL TIMES

OFFSITE HOSPITAL ROUTE

From A – Peterson AFB to

- B Memorial Hospital Central 1400 East Boulder Street Colorado Springs, CO 80909 (719) 365-5000
- **1.** Head northwest on Stewart Ave toward Peterson Blvd Restricted usage road
- **2.** Take the 1st right onto Peterson Blvd Partial restricted usage road
- **3.** Turn left to merge onto E Platte Ave
- 4. Turn right onto Bonfoy Ave
- **5.** Take the 1st left onto E Boulder St Destination will be on the right

Approximately 6.5 miles, 16 minutes travel time.

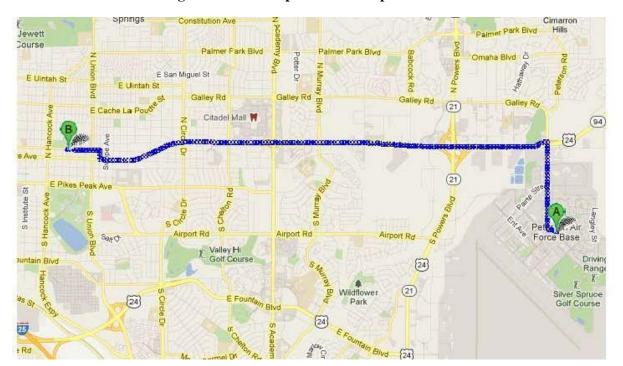


Figure 8-1 Hospital Route Map and Directions

9.0 PRE-ENTRY BRIEFING

The contents and requirements of the SSHP shall be discussed with all site personnel prior initiating site activity, and as part of the general project orientation. Additional discussions on the content of the SSHP will be held as part of the daily tailgate safety meetings. Attendance at all meetings and orientation is required for all personnel, and a sign-in list will be maintained identifying the date, topics discussed, and attendees.

9-1

10.0 EFFECTIVENESS OF PLAN

The effectiveness of this SSHP will be determined by inspections conducted by the Field Task Manager and the SSHO; field observations made related to appropriate contamination control and PPE usage; collection of personal exposure samples and evaluation of the results; comments and suggestions from project employees. All reported hazards will be documented and immediately corrected; suggestions for improvement will be considered for implementation.

10-1



ATTACHMENT A
Activity Hazard Analyses



Environmental Chemical Corporation Activity Hazard Analysis (AHA) – Mobilization and Site Preparation									
Activity/Work Task: Mobilization	T .			RAC) (Use h			M		
Project Location: Peterson Air Force	e Base		Risk Asses	ssment C	Code (RAC)) Matrix			
Contract Number: FA8903-09-R-99	99-R154, TO 0003	G '4			Probabi				
Date Prepared: 09/20/2012		Severity	Frequent	Likely	Occasional	Seldom	Unlike	ly	
Prepared by (Name/Title): Marcus	Johnshov SSHO	Catastrophic	E	E	Н	Н	I	M	
Trepared by (Ivame/True). Iviareus	Johnshoy, 55110	Critical	E	Н	H	M		L	
Reviewed by (Name/Title): Al Nic	kerson, CSP	Marginal	Н	M	M	L		L	
• ` ` ` `		Negligible	M	L Cofety "Control	L	L PAC (See above)		L	
Competent Person (Name/Title):		Probability is the likelihood identified as: Frequent, Like	Step 1: Review each "Hazard" with identified safety "Controls" and determine RAC (See above) Probability is the likelihood to cause an incident, near miss, or accident and identified as: Frequent, Likely, Occasional, Seldom or Unlikely. Severity is the outcome/degree if an incident, near miss, or accident did occur and E = Extremely High F					AC Chart	
Notes: (Field Notes, Review Comm	nents, etc.)	identified as: Catastrophic, Critical, Marginal, or Negligible H = High Risk					ngn rusn		
, ,	,	Step 2: Identify the RAC (Probability/Severity) as E, H, M, or L for each M = Moderate Risk					Risk		
		"Hazard" on AHA. Annota				L = Low Risk		_	
JOB STEPS	POTENTIAL SAFETY / HEALTH HAZARDS	This constitutes the Workplace Hazard Assessment per 20 CFP 1010 132 Additional assessments and 1 R						RAC	
Underground utility survey Identify all overhead utilities	Contact with Overhead Utility Lines – Electrocution, Fires	General Safety - To minimize potential hazards all personnel shall attend site orientation prior to start of work activities in addition to daily Safety Tailgate meetings to familiarize themselves to hazards, emergency procedures, operational aspects and heavy equipment use, and change(s) in site work conditions. Daily housekeeping will be implemented during and at the ends of each workday. Equipment vehicles must be set up with a fire extinguisher (min 10:BC & a First Aid Kit). If drinking water is stored in a cooler, the cooler must be clearly marked "Food & Drink Only – No Samples" Personnel must not work closer than 10 feet from the overhead power line (up to 50 kilovolts) Before work begins, survey the site for overhead power lines. LOOK UP! Never allow equipment or personnel to get closer than 10 feet to an overhead power line. This minimum distance must be increased as the voltage increases. If work must be conducted closer to utilities than guidelines allow, or for placement of insulation, the utility company must be contacted.						M	

	Environme	ental Chemical Corporation	
Act	ivity Hazard Analysis (AHA) – Mobilization and Site Preparation	
	Contact with Overhead Utility Lines – Electrocution, Fires (continued)	 An observer/spotter shall be designated to observe clearance of the equipment and give timely warning for all operations where it is difficult for the operator to maintain the desired clearance by visual means. This shall be the ONLY job the observer is performing when an observer is required. Keep all personnel well away from the equipment whenever it is close to power lines Subsurface utilities must be surveyed and marked prior to intrusive activities. Existing utility markings are not sufficient. Contact an appropriate utility marking company prior to intrusive activities with sufficient time allowed for white-lining and utility marking. ECC personnel will "white-line" or otherwise mark the area where the soil boring activities are to be conducted prior to utility marking. Intrusive soil activities conducted within a five foot "Buffer Zone" (horizontal or vertical, as measured from the outside edge of the utility) of any utility (electric, gas, high pressure, chemical storage tanks, pipelines, sewers, etc.) may require the use of non-aggressive excavation methods such as hand excavation using non-conductive hand tools, use of an air spade, hydro-excavation, or similar means (some jurisdictions require more stringent buffer zones). Spotter will assist the operator/workers to identify unknown conditions during drilling. In some cases, the work location may have to be moved to avoid utilities. If a previously unknown utility line is identified, uncovered, or disturbed during intrusive activities, stop immediately and notify project management and Site Safety and Health Officer (SSHO). Intrusive operations shall not recommence until the line has been evaluated, identified, and the appropriate utility notified. Most utilities and marking services utilize the American Public Works Association Uniform Color Code for marking out utilities. All personnel involved in excavation projects will be familiar with this code: 1. White – Proposed excavation 2. Pi	
	Back Strain or Sprain	 Use proper lifting techniques, move heavy objects with wheelbarrow/carts, seek assistance if items weigh over 50 pounds. 	L
Receipt and placement equipment	Struck-by moving truck	Ensure spotter for trucks and vehicles stays in line-of-sight of driver at all times.	M
vehicle: Spotting of equipment vehicle	Caught in or under equipment vehicle	 Use a spotter to coordinate activities of driver and person setting cribbing, tie-downs, chains. Keep hands out of pinch points. Wear leather work gloves 	M
	Contact with Overhead Utility Lines – Electrocution, Fires	 Personnel must not work closer than 10 feet from the overhead power line (up to 50 kV) Before work begins, survey the site for overhead power lines. LOOK UP! Never allow equipment or personnel to get closer than 10 feet to an overhead power line. This minimum distance must be increased as the voltage increases. If work must be conducted closer to utilities than guidelines allow, or for placement of insulation, the utility company must be contacted. 	М

	Environment	tal Chemical Corporation	
Activ	rity Hazard Analysis (A	HA) – Mobilization and Site Preparation	
	Contact with Overhead Utility Lines – Electrocution, Fires (continued)	 An observer/spotter shall be designated to observe clearance of the equipment and give timely warning for all operations where it is difficult for the operator to maintain the desired clearance by visual means. This shall be the ONLY job the observer is performing when an observer is required. Keep all personnel well away from the equipment whenever it is close to power lines 	
Installation of temporary work zones construction fencing, Installation of erosion controls (as appropriate)	Struck-by hand tools	 Wear leather gloves, safety-glasses, hard hats, safety-toe footwear. Keep hands out of pinch points. Use post driver, not sledge hammer for placing fence posts, tape top of fence post or install mushroom caps to reduce potential for scrapes. 	L
	Sprains/strains Struck-By (hand tools)	Use two people to carry heavy loads of fencing/posts. Do not lift and carry more than comfortable weight for individual, 50 pounds max.	L
Establishment of work zones, decontamination stations for personnel	Slips/trips/falls	 Wear high traction safety-toe footwear. Keep loads manageable to not obstruct vision. 	L
and equipment	Scrapes and cuts	Wear safety glasses, gloves and long sleeves.	L
and equipment	Contact with poisonous plants (e.g. poison ivy)	 Inspect area before starting Wear long sleeve shirts, tuck sleeves and pant legs. If there is heavy growth, wear disposable coveralls and use barrier cream, e.g., Ivy Block. Have Tecnu or other poison ivy cleanser on hand, and wash immediately after contact. 	M
	Stung by bees/hornets, bit by ticks or snakes	 Inspect areas for hives. Ensure allergic individuals have emergency medical kit and are committed to using it. Use insect repellant containing DEET on exposed skin and Permethrin on clothing. Do not approach snakes. If bitten, seek medical attention. 	M
	Struck by moving equipment	Personnel will stay out of equipment swing areas and pinch-points. Personnel must wear high visibility garments when working around moving equipment and/or traffic.	L
	Fire/explosion of gasoline	 Allow equipment to cool before refueling, and eliminate other sources of ignition. Use only approved National Fire Protection Agency (NFPA) safety cans for gasoline. Cleanup spills immediately. 	L
	Heat Stress	Rest/work cycles, fluids, and temperature monitoring. Follow ECC SO ESQ-8.5 Evaluate weather conditions as to heat stress while wearing protective clothing while decontaminating equipment.	M
	Eye injuries	Safety glasses with side shields (impact resistant)	L
Stop work and notify the Team Leader if you are not sure how to perform your task safely!	Stop work and notify the Team Leader if you are not sure how to perform your task safely!	Stop work and notify the Team Leader if you are not sure how to perform your task safely!	

Equipment to be Used	Training Requirements	Inspection Requirements
 Field vehicle, Drill rig, Hand tools Support Zone Cell phone communication Eyewash station Fire extinguishers First aid kit Drinking water Spill containment supplies Air Monitoring equipment Decontamination supplies 	 Only qualified operators permitted to operate mobile equipment. First Aid/Cardiopulmonary Resuscitation training (at least two individuals onsite) Initial Safety Orientation Daily Safety Tailgate Meetings Emergency Response Plan Current Hazardous Waste Operations and Emergency Response (HAZWOPER) training 	 Equipment - Receipt and inspected by SSHO. Daily equipment inspection by operator Weekly inspection of Fire Extinguishers and First Aid Kits. Daily inspection of hand and power tools with replacement of damaged items.

Activity Hazard Analysis Training Log

Activity/Phase of Work: Mobilization and Site Preparation

By signing below: I agree to follow the work steps and implement the hazard controls. I agree to stop work when conditions or hazards change, when work cannot be performed as written, or when instructions become unclear during execution. I am qualified and fit to perform the work.

PRINT NAME	SIGNATURE	EMPLOYER	DATE

Act	Env tivity Hazard Anal			l Corporati Push Drillir		oil Samnling	
Activity/Work Task: Direct	<u> </u>	<u> </u>		ssessment Code		<u> </u>	M
Project Location: Peterson	AFB		Risk	Assessment	Code (RA	AC) Matrix	
Contract Number: FA8903-0	9-R-9999-R154, TO 0003	Severity			Probab	oility	
Date Prepared: 09/20/2012		Severity	Frequent	Likely	Occasional	Seldom	Unlikely
Prepared by (Name/Title):	Marcus Johnshoy, SSHO	Catastrophic Critical	E E	E H	H H	H M	M L
Reviewed by (Name/Title):	Al Nickerson, CSP	Marginal Negligible	H M	M L	M L	L L	L L
Competent Person (Name/Title): Qualified Driller to be identified Notes: (Field Notes, Review Comments, etc.)		Step 1: Review each "Hazard" with identified safety "Controls" and determine RAC (See above) Probability is the likelihood to cause an incident, near miss, or accident and identified as: Frequent, Likely, Occasional, Seldom or Unlikely. Severity is the outcome/degree if an incident, near miss, or accident did occur and identified as: Catastrophic, Critical, Marginal, or Negligible Step 2: Identify the RAC (Probability/Severity) as E, H, M, or L for each "Hazard" on AHA. Annotate the overall highest RAC at the top of AHA. L = Low Risk					
JOB STEPS	POTENTIAL SAFETY / HEALTH HAZARDS	RECOMMENDED CONTROLS Note: Standard PPE required for this activity includes Hard Hat, Safety glasses with side protection, and safety-toe footwear. Additional PPE requirements are listed in this column depending on the hazard. This constitutes the Workplace Hazard Assessment per 29 CFR 1910.132. Additional assessments and PPE selection when needed will be documented on a daily briefing sign-in form and signed by the SSHS in accordance with ECC SOP ESQ 6.1. Hazard assessment and					n a RAC
Mobilization of Equipment and Supplies	Slip/Trip/Falls	Work areas and Even terrain was	d means of access s ill be utilized as un	hall be maintained sa loading areas.	fe and orderly.	Protection Plan, as required. red or clearly identified.	L
	Vehicular Traffic		e used when backin	g up trucks and movi thing will be worn w	ng equipment.	ar roadways and moving equipme	M nt.
	Back Injuries	Site personnelMechanical de	will be instructed o	n proper lifting techn d to reduce manual h f mechanical devices.	andling of mate	rials.	M
	Dropped Objects	Steel toe boot	ts meeting America	n National Standards	Institute (ANS)	I) Standard Z41 will be worn.	L
	Overhead Hazards Eye Injury		-	hard hats that meet Andard Z87 will be wo		Z89.1.	L L

		vironmental Chemical Corporation	
Act	tivity Hazard Ana	alysis (AHA) – Direct-Push Drilling and Soil Sampling	
Mobilization of Equipment and Supplies (continued)	Struck By/Against	 Personnel will understand and review hand signals. All machines will be equipped with backup alarms. 	M
Coring through concrete and/or	Underground Utility Strikes	Ensure all location have been surveyed and properly marked for utilities.	M
asphalt	Struck by equipment Personal injury	 Follow manufacturer's instruction for equipment operation. Starting coring equipment with cutting blades in the hole may cause violent lurching of equipment. If possible, allow cutter to rotate freely before initiating coring activities. 	L
Direct-push Fueling	Fire	 All fuel tank/trucks shall be grounded during fueling operations. Smoking and open flames are not permitted within 50 feet of fueling/greasing areas. All equipment shall be equipped with 10 pound (lb.) ABC type fire extinguishers. 10-lb. ABC type fire extinguishers shall be readily available during fuel/greasing operations. 	M
	Chemical Exposure	• Protective clothing (i.e., chemical gloves and safety glasses) will be worn during fueling operations. Skin will be rinsed with water if contact with hazardous material occurs.	L
	Spills	 Spill and absorbent materials will be readily available. Fueling operations shall be constantly monitored. Employees will be instructed as to proper fueling techniques. Dispensing nozzles shall be an approved automatic-closing type without a latch-open device. Fuel nozzle and hose will be secured in holder after use. Fuel caps will be secured after fueling operations. Fuel tanks and equipment will be grounded and bonded during fueling operations. 	L
Direct-push Set-Up	Rollovers	Equipment shall be set-up on stable ground and maintained level. Cribbing will be used when necessary. Outriggers shall be extended per the manufacturer's specification.	L
	Back Injuries	 Site personnel will be instructed on proper lifting techniques. Mechanical devices will be utilized to reduce manual handling of materials. Team lifting will be used in lieu of mechanical devices. 	M
	Overhead Hazards	 All personnel will wear hard hats. All ropes will be rated for the load in which it is expected to lift. All ropes will be inspected at the beginning of each work shift. All ground personnel will stay clear of all suspended loads. All equipment will stay a minimum of 15 feet from energized electrical lines. This distance will increase as the voltage of the power lines increase. 	L
	Slip/Trip/Falls	 Work areas and means of access shall be maintained safe and orderly. Even terrain will be utilized as unloading areas. Tripping and poor footing hazards will be repaired as they are discovered or clearly identified. 	L
	Dropped Objects	Steel toe boots meeting ANSI Standard Z41 will be worn.	L
	Eye Injury	Safety glasses meeting ANSI Standard Z87 will be worn.	L
Direct-push Operations	Struck By/Against	 EMERGENCY KILL SWITCH SHALL BE TESTED EACH DAY BEFORE OPERATION. No loose clothing, gauntlet-type gloves, rings or watches will be worn by personnel operating direct push equipment. Auger guides will be used on hard surfaces. 	L

	Environmental Chemical Corporation				
Act	tivity Hazard Ana	alysis (AHA) – Direct-Push Drilling and Soil Sampling			
Direct-push Operations (continued)		 Personnel will understand and review hand signals. Direct push and support equipment will be equipped with backup alarms. The direct push operator will verbally alert employees and visually ensure employees are clear from dangerous parts of the equipment prior to starting or engaging equipment. All direct push equipment shall be equipped with emergency shut off devices. Internal combustion engines will be equipped with an ignition or grounding switch. Diesel engines will be equipped with quick closing valves which will shut off air to the intake manifold. Electric motors will be equipped with suitable switch in motor circuits. Gears will be enclosed by a well-constructed metal guard and securely installed. Guards shall be maintained in place when machinery is in use. 			
	Underground Hazards	All underground utilities will be identified and marked by an appropriate agency prior to drilling.	M		
	Flying Objects and Debris	 Splash shields and chemical goggles meeting ANSI Standard Z87 will be worn where applicable. A portable eye wash station will be located adjacent to the work area. 	L		
	Overhead Hazards	 All personnel will wear hard hats meeting ANSI Standard Z89.1. All ropes will be rated for the load in which it is expected to lift. All ropes will be inspected at the beginning of each work shift. All ground personnel will stay clear of all suspended loads. All equipment will stay a minimum of 10 feet from energized electrical lines. This distance will increase as the voltage of the power lines increase. 	L		
	Chemical Exposure	 Personal exposure monitoring for contaminants of concern is not anticipated. The primary concern is protection from dermal contact. Exclusion zone areas will be identified. Personnel Protective Equipment (PPE) will be worn as specified in Site Safety and Health Plan (SSHP). Skin will be rinsed with water if contact with hazardous materials occurs. 	L		
	Noise	All equipment will be equipped with manufacturers required mufflers. Noise monitoring will be conducted. Hearing protection will be worn when noise exposures exceed 85 dBA slow.	L		
	Fire	 Smoking and open flames are not permitted in the Exclusion Zone. All equipment shall be equipped with readily available 10-lb. ABC type fire extinguishers. 	L		
	Slip/Trip/Falls	 Personnel will clear walkways of equipment and materials. Other obstructions will be marked, identified or barricaded. Tripping and poor footing hazards will be repaired as discovered or will be clearly identified. Debris will not be allowed to accumulate where it becomes a hazard. When repairing drill rig, personnel will utilize fall protection when 6 feet above ground. 	L		
	Back Injuries	 Site personnel will be instructed on proper lifting techniques. Mechanical devices will be utilized to reduce manual material handling. Team lifting will be utilized in lieu of mechanical devices. 	L		

	Er	nvironmental Chemical Corporation						
A		alysis (AHA) – Direct-Push Drilling and Soil Sampling						
Direct-push Operations (continued)	Sharp Objects	 Cut resistant work gloves will be worn. All hand and power tools will be maintained in safe condition. First aid kits will be available by work area. 	L					
Soil Sampling	Exposure to Contaminants	 Exclusion zone areas will be identified. PPE will be worn as specified in SSHP. Inhalation exposures to soil chemicals of concern are not expected, although sampling activities should be conducted upwind as possible. Primary concern is the prevention of dermal contact. Skin will be rinsed with water if contact with hazardous materials occurs. All equipment and materials will be decontaminated as stated in the SSHP and Work Plan. 	L					
	Eye Injury	 Safety glasses that meet ANSI Standard Z87 will be worn. Portable eye wash station will be available. 						
	Hand lacerations cutting open plastic sample tubes	 Utilize a tool other than a razor knife. Wear leather or Kevlar work gloves. 	L					
	Overhead Hazards	Personnel will be required to wear hard hats meeting ANSI Standard Z89.1. ANSI Standard Z89.1.	L					
	Dropped Objects Fire	 Steel toe boots meeting ANSI Standard Z41 will be worn. Smoking is not permitted in CRZ or exclusion zone areas. 10-lb. ABC type fire extinguishers will be located adjacent to the work area. 	L L					
Equipment Repair	Fire	10-lb. ABC type fire extinguisher will be located adjacent to the work area. Smoking will not be allowed in the work area. A Hot Work Permit shall be obtained prior to any welding, cutting, or grinding	L					
	Struck By	 All direct push equipment shall be equipped with emergency shut off devices. Internal combustion engines will be equipped with an ignition or grounding switch. Diesel engines will be equipped with quick closing valves which will shut off air to the intake manifold. Electric motors will be equipped with suitable switch in motor circuits. No work will be performed on engines, motors, hoists, etc. until it has been properly locked and tagged out. After clutches have been disengaged, the bypass valve should be opened to divert air from the clutches. Machinery will not be lubricated while it is in operation or running. 	M					
	Sharp Objects – punctures, abrasions	 Cut resistant work gloves will be worn. All hand and power tools will be maintained in safe condition. First aid kits will be available by work area. Guards will be kept in place while using hand tools 	L					
	Overhead Hazards	Personnel are required to wear hard hats.	L					
	Dropped Objects/foot injury	Steel toe boots meeting ANSI Standard Z41 will be worn.	L					
	Flying Objects and Debris	Safety glasses meeting ANSI Standard Z87 will be worn.	L					
	Chemical Exposure	 Protective clothing (i.e., chemical gloves and safety glasses) will be worn were applicable. Skin will be rinsed with water if contact with hazardous material occurs. 	L					

Environmental Chemical Corporation					
Activity Hazard Analysis (AHA) – Direct-Push Drilling and Soil Sampling					
Equipment Repair (continued)	Slip/Trip/Fall		 Personnel will clear walkways of equipment at Other obstructions will be marked, identified of Tripping and poor footing hazards will be repaired. 		L
Stop work and notify the Team Leader if you are not sure how to perform your task safely!	Stop work and notify the Leader if you are not sur perform your task safely	re how to	Stop work and notify the Team Leader if you are not sur	re how to perform your task safely!	
E					

Equipment to be Used	Training Requirements	Inspection Requirements
 Direct push Fire Extinguishers, air monitoring equipment, noise meter, First Aid kits Spill control materials Chains, ropes, and/or slings Concrete/asphalt coring equipment 	 Only qualified drill rig operator permitted to operate Direct push and coring equipment First Aid/Cardiopulmonary Resuscitation training (at least two individuals onsite) Initial Safety Orientation Daily Safety Tailgate Meetings Emergency Response Plan Current HAZWOPER training 	 Equipment - Receipt and inspected by the Site Safety and Health Officer (SSHO). Daily drill rig and coring equipment inspection by operator – see attached checklist for direct push equipment. Weekly inspection of Fire Extinguishers and First Aid Kits. Daily inspection of hand and power tools with replacement of damaged items.

Activity Hazard Analysis Training Log

Activity/Phase of Work: Direct-push Use and Soil Sampling

By signing below: I agree to follow the work steps and implement the hazard controls. I agree to stop work when conditions or hazards change, when work cannot be performed as written, or when instructions become unclear during execution. I am qualified and fit to perform the work.

PRINT NAME	SIGNATURE	EMPLOYER	DATE

ECC

DIRECT-PUSH / DIRECT-PUSH INSPECTION CHECKLIST

Contractor:	Rig Type:	Rig Number:	Date:		
Inspected by:	Project:				
(✓) If OK	(-) Not Applicable (2	X) If Correction Require	ed		
I. Rig Carrier		Control Batteries			
() Overall Appearance	() Remote Control Receiver Batteries				
() Oil Leaks	() Tie-Down Anchors				
() Fuel Leaks	() Ignition Switch				
() Fire Extinguisher	() Hydraulic System Safety Shut-Off				
() Exhaust System	() Other:				
() Outrigger Jacks	() Other:				
() Drive Chains	` ,				
() Tires	IV. Down hole Equipment				
() Suspension		() Drill pipe			
() Tie-Down Straps	() Drill col				
() Tie-Down Anchors	() Core Ro				
() Other:	() Core Bar				
() Other:	() Sampler				
	() Other:				
II. Mast	() Other:				
() Mast Rams/Cylinders					
() Hinge Pins	V. Miscella	neous Safety Items			
() Fasteners (Nuts, Bolts)	() <i>KILL SV</i>	<u> VITCH – FÜNCTIONAL</u>	AND ACCESSIBLE		
() Winch	() Placards	/Warning Signs			
() Cable	() First Aid	l Kit			
() Lifting Hook and Safety Lat	tch () Applicat	ole Regulation Posting			
() Cable Clamps	() Material	Safety Data Sheets			
() Other:	() Eye Was	sh			
() Other:	() Fire Exti	nguisher			
					
III. Rig Engine(s)	() Other:				
() Fuel Tank(s)					
() Exhaust System		al Protective Equipment			
() Electrical System	() Hard Ha				
() Belt/Drive Guards	() Safety G				
() Engine Compartment Cover					
() System Temperature	() Hearing				
() Fluid Filler Caps Secure	() Hand Pro				
() Emergency Shut-down Syst		Tick Protection)			
() Heat Shields					
() Fluid Leaks	() Other:				
() Gauges	**** * •				
() Clutches	VII. Licens				
() Other:	() Drillers 1				
() Other:	() Hydrauli	ic license			
IV. Drill Unit	VIII Hand	Power Tools			
() Fluid Leaks					
() Hydraulic Hoses					
() Gauges () Tubing Cutter					
() Loose Bolts		() Extension Cords			
() Tracks	() Other:				
() -14010	() Guici				